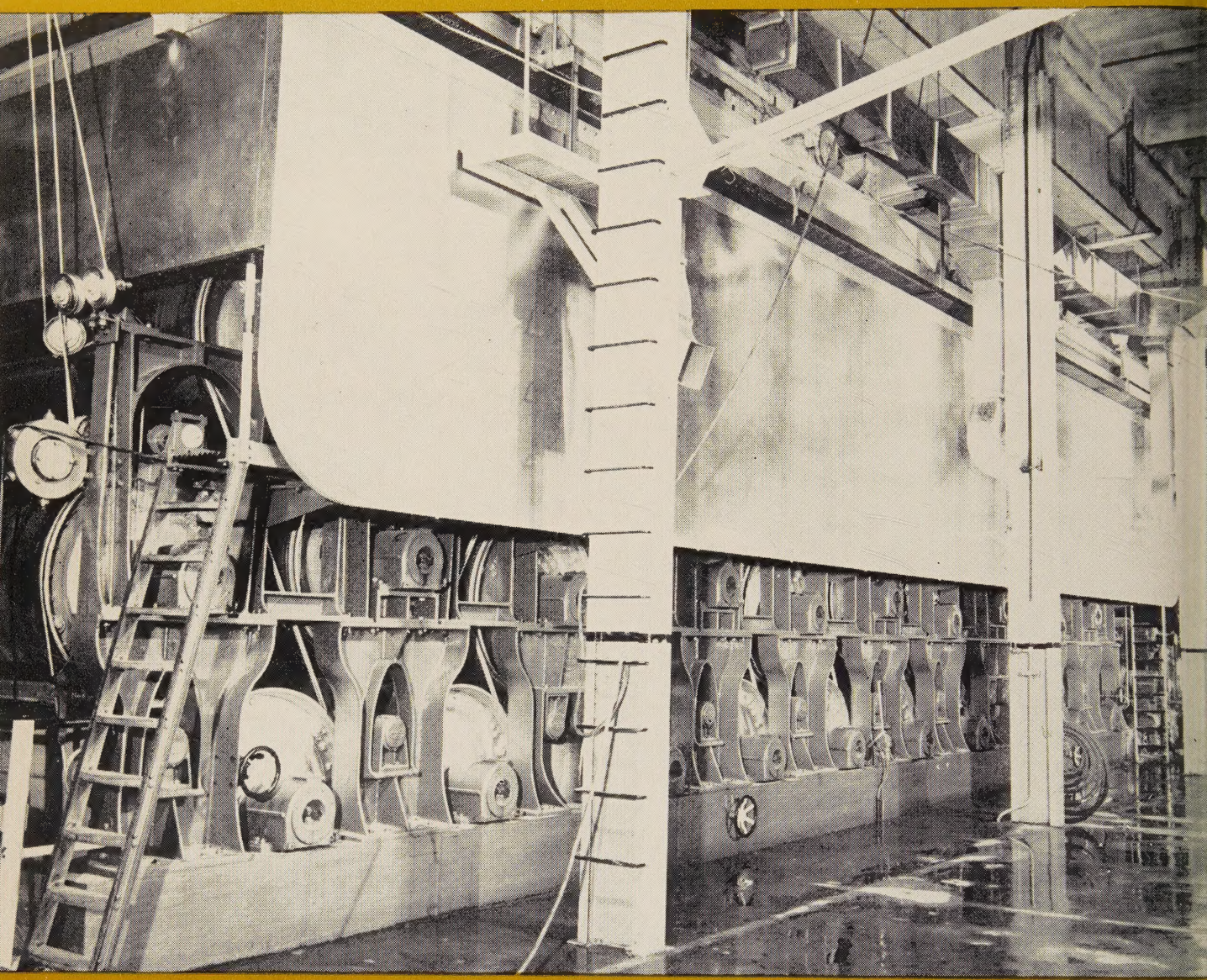
Tappi

PUBLISHED BY THE TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY

FEBRUARY 1951

MOST MODERN CYLINDER BOARD MACHINE IN CANADA



Designed, built and installed for Anglo-Canadian Pulp and Paper Mills Ltd., Quebec City by . . .

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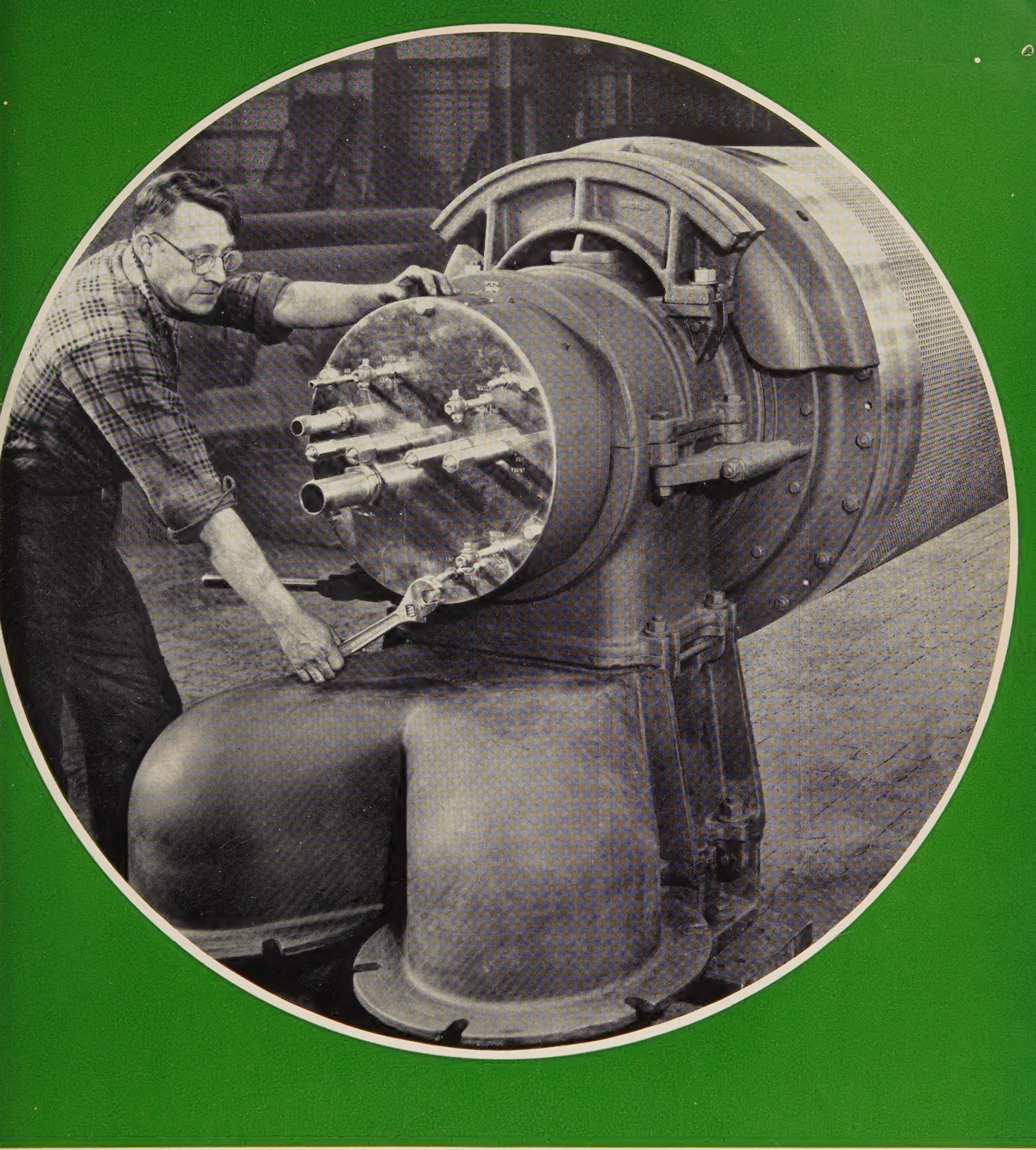

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Beloit Suction Couch Roll (44″ diameter), ready for shipment to a Finnish mill, has a drilling pattern "tailor-made" for the specific application. Position of each end deckle and of the dual suction chamber can be adjusted while in operation. Lubrication of the heavy-duty front main bearing and back internal bearing is conveniently handled from the front of the roll.—*Beloit Iron Works, Beloit, Wisconsin.*

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PAPER MACHINERY

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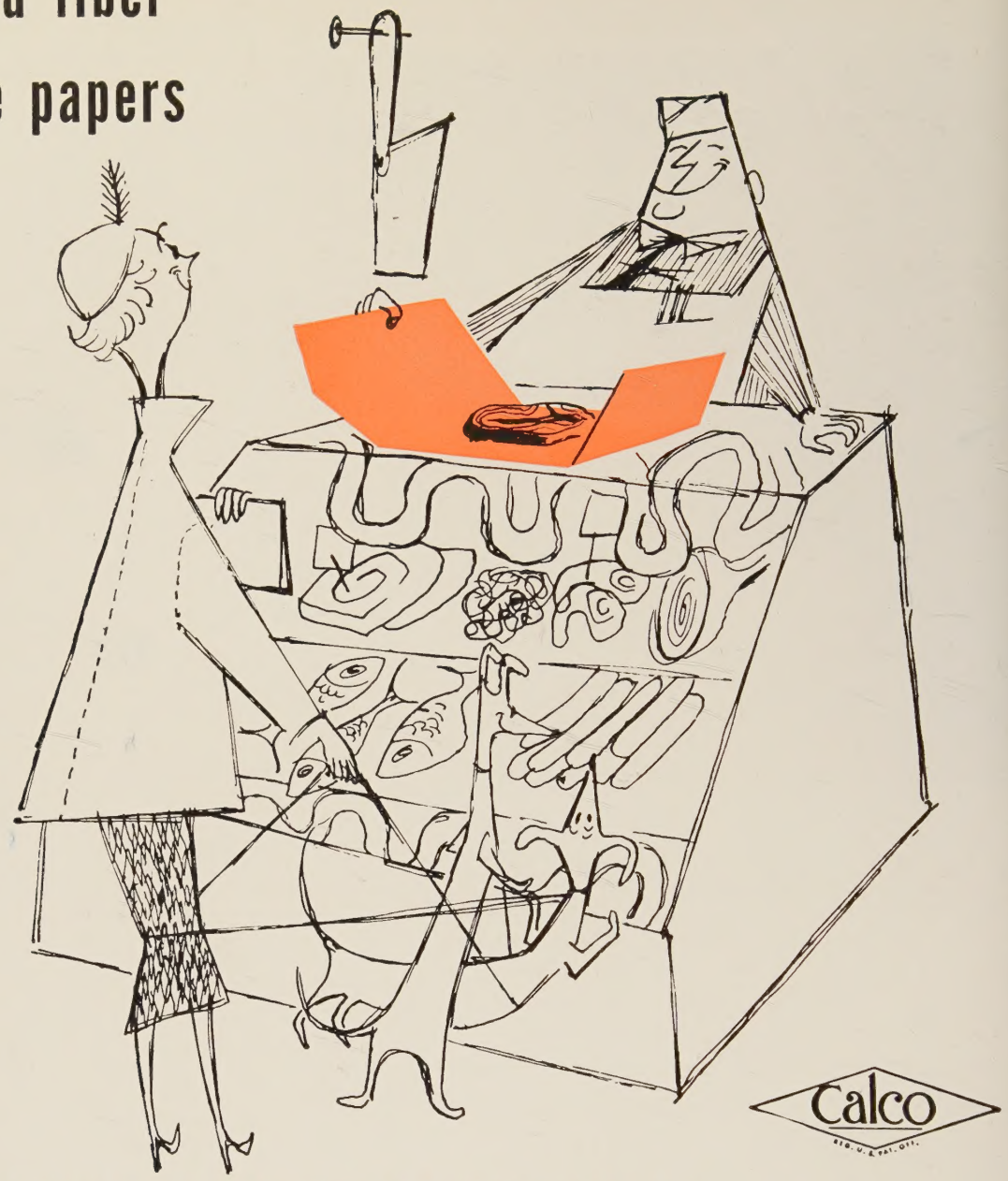

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Excellent solubility, good tinctorial value and fastness to light, acid, silicate and alkali make Calco PHENO FAST SCARLET 4BNC one of the finest and brightest of the Direct Scarlets.

PHENO FAST SCARLET 4BNC imparts good color to many types of red papers, and it is especially recommended for producing red fiber for granite papers such as butcher's wrap. It is suitable for coloring papers which are to be treated with melamine wet-strength resins.

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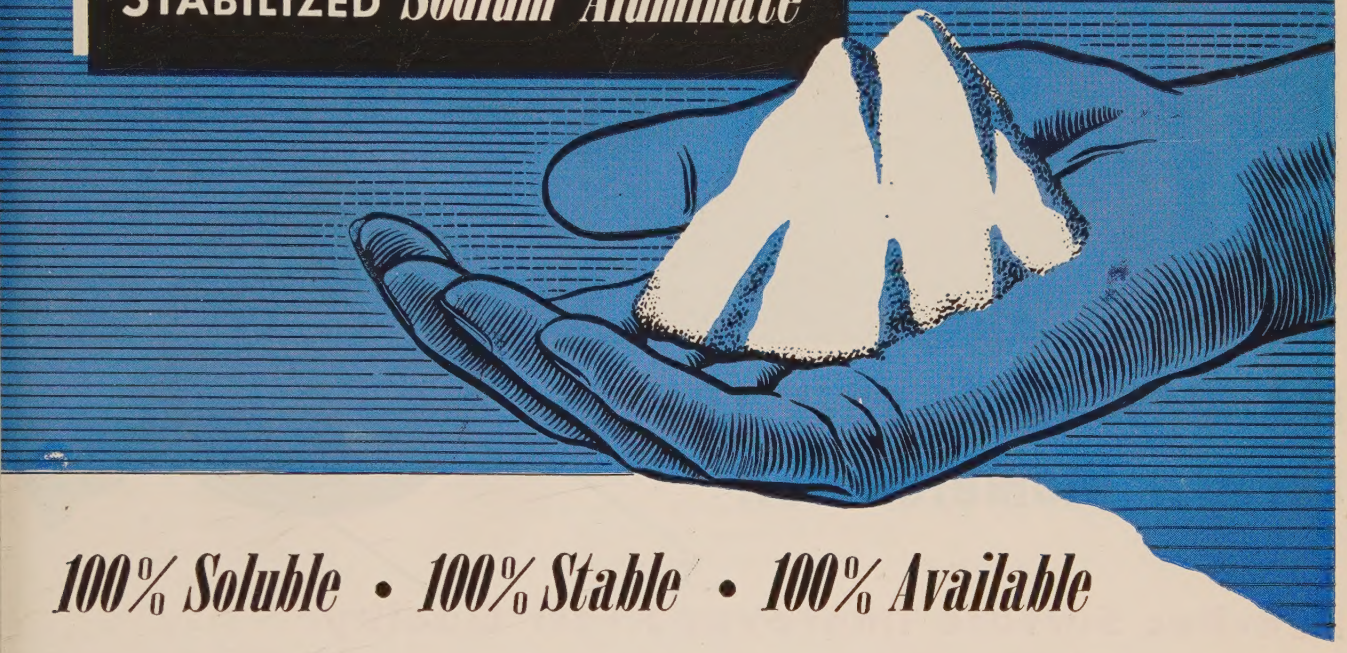

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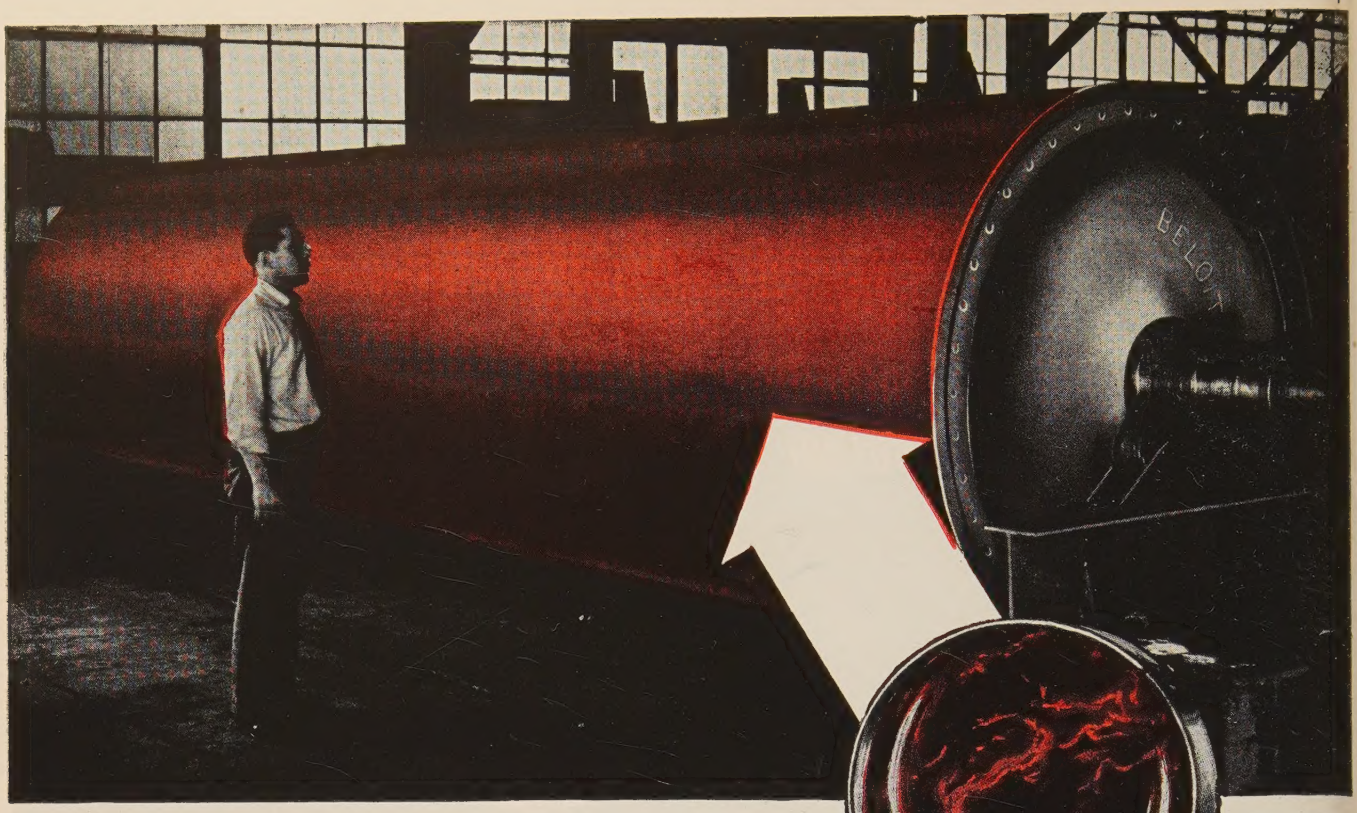

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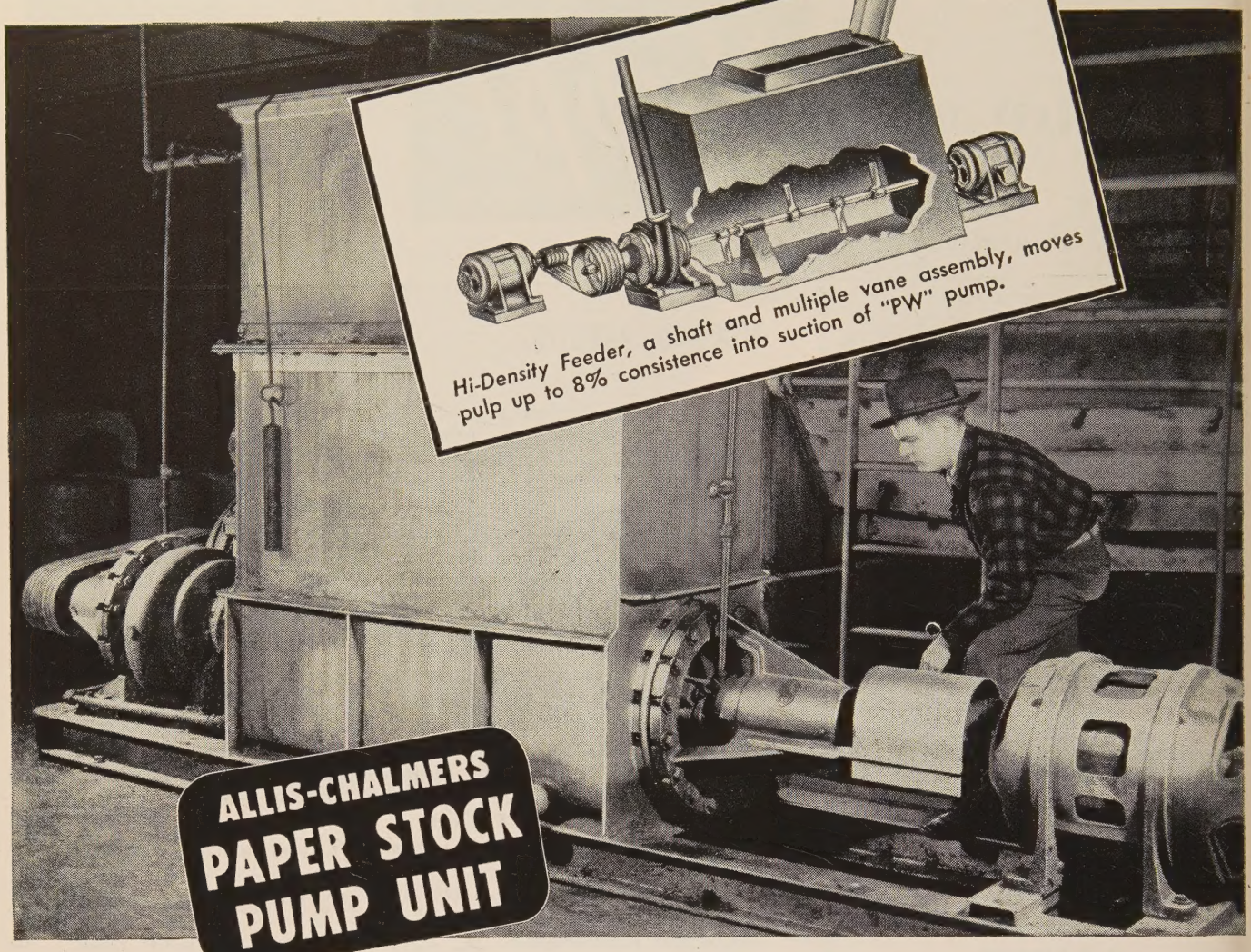
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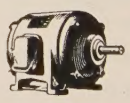
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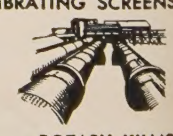
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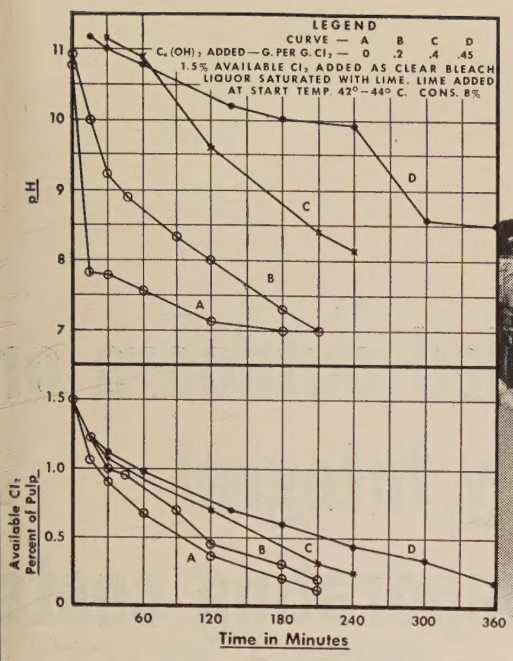
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CURVE — A B C D
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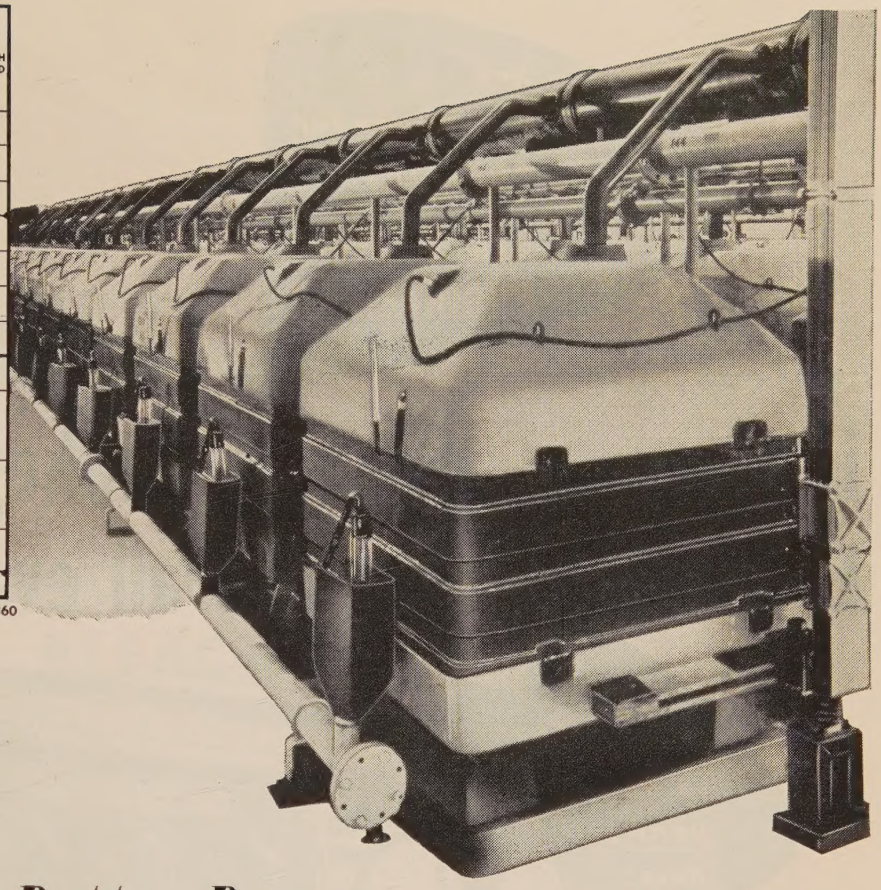
1.5% AVAILABLE Cl_2 ADDED AS CLEAR BLEACH LIQUOR SATURATED WITH LIME. LIME ADDED AT START TEMP. 42°–44° C. CONS. 8%
pH
Available Cl₂ Percent of Pulp
Time in Minutes
60 120 180 240 300 360

Effect of lime additions on pH and rate of chlorine consumption in a hypochlorite reaction with chlorinated sulfate pulp.



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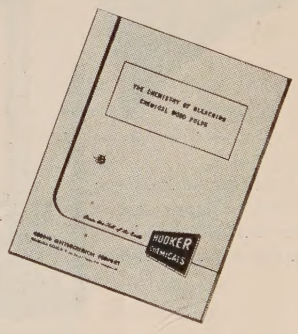

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- 201 Process and Equipment for Making Bleach Liquor for Use Without Settling
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- 242 Production and Use of Unsettled Bleach Liquor
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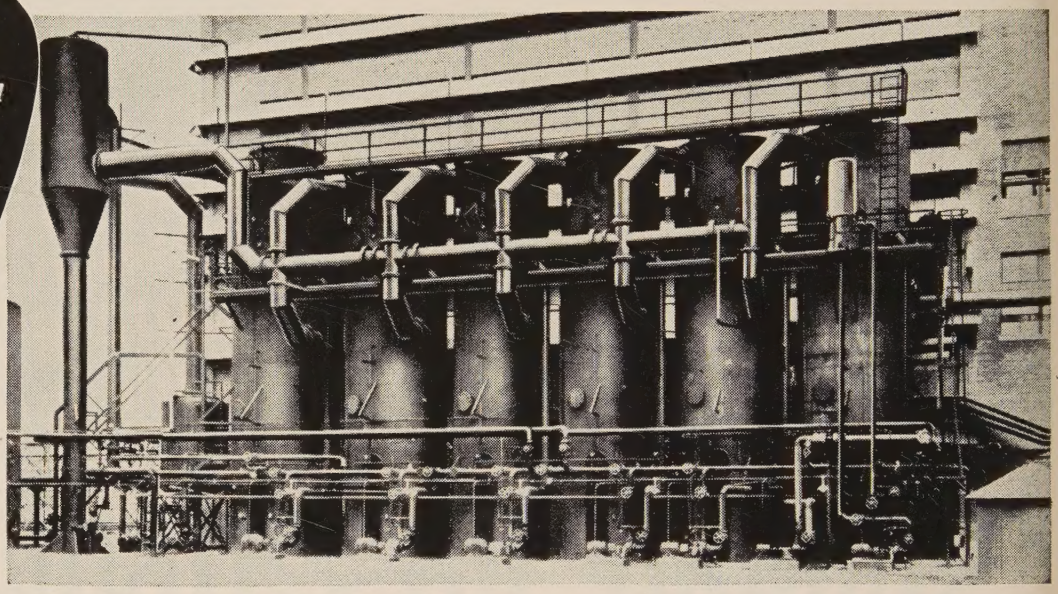

Conkey Integral Sextuple Effect Evaporator outdoor installation at southern pulp mill.

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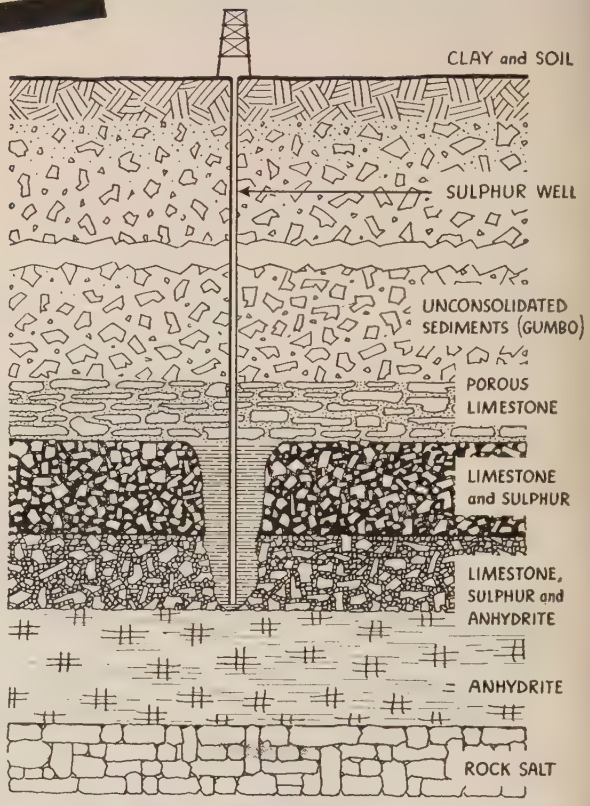

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PAPER PRODUCT 2142, a sizing agent, increases the water-repellency of asbestos shingles.

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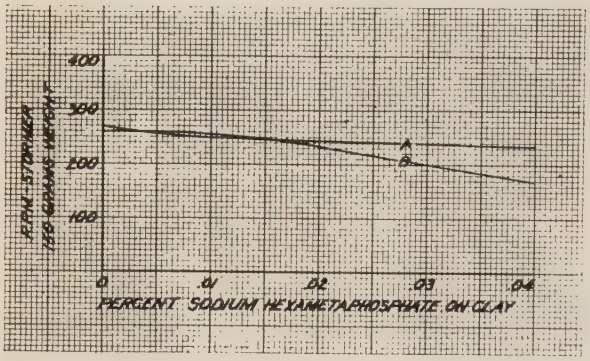


Effect of Sodium Hexametaphosphate on Flow Characteristics of Clay-Casein Colors. (Graph Shows Two Typical Medium Fine Clays.)

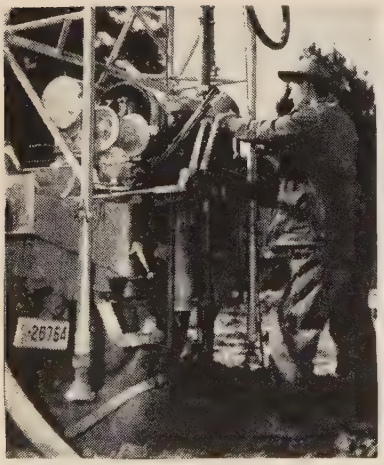
EFFECT OF DISPERSING AGENTS IN CLAY-CASEIN MIXTURES In contrast to the effect observed in clay-water and clay-starch systems, the use of dispersing agents in clay-casein mixtures produces no increase in fluidity. If used in excessive amounts such agents are likely to result in a harmful effect. The accompanying graph shows the effect of increasing amounts of sodium hexametaphosphate on the flow characteristics of casein systems. The formula consisted of 42% total solids with 15 parts of casein to 100 parts of clay.

TESTING CLAY ABRASIVENESS The widest difference in abrasiveness occurs in filler grade clays, hence it is important to check this factor regularly. Years ago water washed filler clays gave superior abrasion results and a number of paper mills still favor them for that reason. However, the technique of refining Huber air-floated filler clays has advanced so far in recent years that there is now essentially no difference between the two types. Abrasiveness is measured by circulating a clay-water slurry through a perforated rubbing block which contacts a small section of Fourdrinier wire. Abrasiveness of the clay is recorded in terms of loss in wire weight in milligrams. Actual testing procedure varies in different paper mills, yielding different numerical values, but the relationship between a given set of clays should hold regardless of procedure used.

CLAY	Abrasion Test* (Wire Loss)
Huber Aerfloted Hi-White (Ga.)	15 mg.
Huber Aerfloted Paragon (S.C.)	16
Huber Water Washed Filler (Ga.)	22
Water Washed Filler "A"	28
Water Washed Filler "B"	22

* by the Valley Iron Works abrasion tester. Absolute values vary depending on test method used.

NOTE: Samples "A" and "B" are competitive clays obtained from mills using them regularly and are believed to be representative.



Core drilling a test hole at Huber's Langley, S. C. mine.

VARIATIONS IN CLAY DEPOSITS The kaolin clay deposits of Georgia and South Carolina vary in such essential properties as particle size, color and particularly in their rheological (flow) behavior. Such variations exist not only between deposits several miles apart, but often within short distances in individual deposits. The characteristics of a commercial-grade clay reflect, with slight modification, the nature of the crude from which it originates, hence it is important to predetermine the quality and uniformity of a deposit. This is done by drilling test holes which serve not only to secure samples for analysis but also to determine the boundaries of the clay areas.

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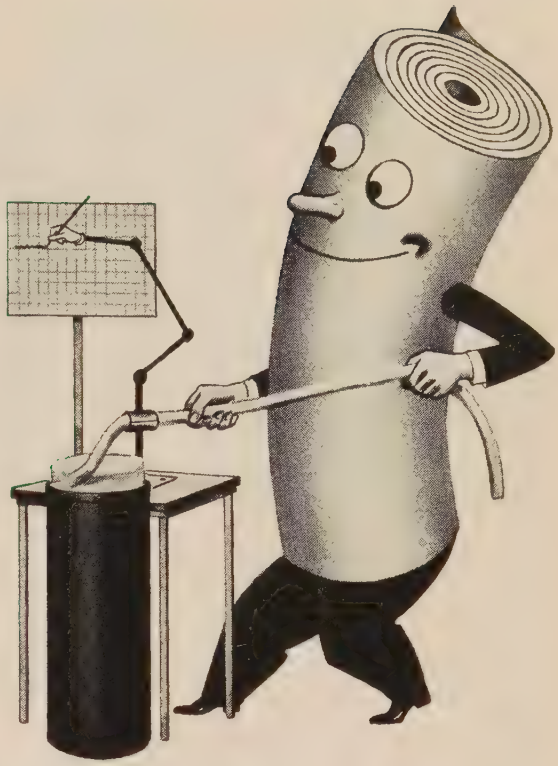

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Operating Principles as Related to Quality Control

A. E. BACHMANN

QUALITY control is a problem that we all have in common. To prevent irritating our customers with even small or infrequent doses of defective paper is the prime manufacturing responsibility of all of us. We must, above all else, completely close the door of the mill to shipping defective paper of any type that could cause our customers waste or defective products. We must arrange sampling and inspection methods to detect even slight amounts of defects which would, if allowed to remain, contaminate the whole shipment. Our customers use all the paper and even if only small amounts of defective paper are shipped, they will find it in their processing of the paper. In most cases a customer will be just as irritated by a small amount as by a larger amount because he constantly expects more of it and may run the entire lot at a slower production speed as the result of running into it. Also in many cases it only takes a single defect of some types to break a plate and cause him extended delays and extra cost.

Before the advent of modern technical control, much quality control consisted merely of running our orders to constant conditions of furnish ingredients and constant standard machine settings of operational equipment. Occasionally, even in this relatively enlightened age we all, I am sure, resort to this procedure when confronted by complaints which our control system is inadequate to resolve for us. In the absence of a completely adequate control system our efforts at control, at times, have consisted of personally holding our hand to the leaking dike, in the form of continuous trouble shooting. Some of our efforts of the past, at personal supervision, might even be likened to the fullback of a football team backing up the line and making the tackles caused by holes in the line of quality controls in front of him. The need for effective supervision or backing up the line of controls will always be present but obviously if the line of controls is so weak that the backfield of successive layers of the line organization must continually back it up, we as members of the management coaching staff might well devote more of our time to developing a new defense in the form of a more effective line of controls, to the end that our front-line supervisors' efforts will be reduced to stopping only an occasional break-through.

Quality control is probably our most important single problem because it is a necessary precedent to all our various cost controls such as waste control, production control, cost control, and budgetary control. It forms essentially the first basic step in cost control.

Without an effective system of quality control it is quite useless to consider making any cost reduction in the form of increased speeds or elimination of excess materials. In the absence of adequate quality control naturally much waste will be made as the result of not speedily detecting off-quality paper. Because of complaints or rejections speeds may be held down and unnecessary extra materials may be used as an additional safety factor for quality. Whenever the slightest doubt regarding quality exists all cost reduction efforts are usually speedily abandoned. On the other hand one of our best opportunities for cost reduction lies in attracting extra sales volume and this results largely from establishing a reputation for making uniformly good deliveries. Obviously, therefore, quality control forms the very bottom layer in a pyramid of controls through which the success of the entire enterprise is effectuated.

Before discussing quality control in any detail we might first examine briefly this pyramid of controls to place quality control in its proper perspective as a basic control out of which all other controls develop.

Control is a very much overworked and all inclusive word but it nevertheless does have a connotation for us, which is not exactly expressed by any other word, so we therefore still continue to use it for this purpose. The word "control" suggests that all is under control; that the routine at all times goes along satisfactorily at any level of standards; that we by some legerdemain can bring about that happy condition where we have only to examine a few reports and push a few buttons to fix anything that gets out of line with our quality or cost. This concept I suspect is much like the search for "truth," which has been defined as "something that temporarily has the appearance of permanence." Control also is something that temporarily has the appearance of permanence. In other words, we will never realize it fully but we can, however, approach it to a high degree. The development of even a fairly satisfactory degree of control is a long process. It consists essentially of many little things, none of which of themselves are especially important, but the aggregate of which is extremely important. To attain control we must in effect get the routine established on a mistake-proof basis, with arrangements for improving the existing routine in progressive steps to the ultimate and at the same time controlling the progressive improvements as they, each in turn, become standard.

The need for control stems largely from the universally recognized principle of organization that ultimate responsibility in the form of accountability cannot be delegated. Hence a delegant in order to be able to fulfill his accountability to his own superior must establish a system of control to aid in supervision before he can safely delegate responsibility.

Control then is a system or a technique by which a delegant at various organizational levels is informed of an impending or past default of a delegated or even subdelegated responsibility in terms of who, where, what, when, why, and how much. The system or technique used naturally is different, particularly in detail, at various organizational levels. Essentially separate but coordinated systems must be devised for

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control of the continuity of a business at administrative levels, profits at general management levels, costs at production management levels, operation at superintendence levels, and routine at foremen levels. The techniques of control presently available for these respective levels are the breakeven point analysis, budgets, standard costs, standards, records, standard practise, and signposts. In quality control we are concerned primarily with the bottom level—standards, records, standard practise, and signposts.

Control of any kind, including quality control, then consists of a method of action or a series of steps through which the prescribed policies or purposes of the organization will be at all times accomplished. To protect our system from the ravages of inevitable human frailties, we need to build it up as a set of signposts which will function almost automatically to preclude the possibility for any individual himself or any of his subordinates, even when following the path of least resistance, to fail to have instantly available all the relevant facts and tools which will literally prompt him to the proper and effective discharge of his particular duties or responsibilities. We need further to develop a system of report cards in the form of suitable statistics records which evaluates in terms of some standard the effectiveness with which each individual in the organization uses these signposts, and also, that the signposts themselves can be evaluated for their effectiveness in controlling the quality.

Among such signposts which can be used effectively for quality control are adequately designed forms, reports, recaps, samples of various kinds, instruction cards, standard procedures, trouble shooting procedures, grade packages, complaint letters, complaint signs, broke logs, quality logs, etc.

Complete quality control includes the liberal use of such signposts arranged in order of logical steps designed to translate some of the principles previously discussed into more practical terms. To do this we must arrange to gather on a routine basis many types of samples, design logs, or forms for recording the results of all operations, install a testing station with testers, and also make provision for the assigning of specific inspection responsibilities for specific qualities or defects which are not measurable, to various members of the machine crews.

Since the control system envisages control at constantly increasing levels of quality and constantly decreasing levels of cost, it becomes necessary also to provide for the control research function. Such research must be distinguished from fundamental research organizationally. Such applied research consists of trouble shooting, complaint investigation, and research in connection with complaints. It also comprises research on elimination of defects as disclosed by nonstandard paper lists and investigations of differences disclosed between runs of extra good properties and ordinary ones. Also provision must be made for making trials together with tests on possible material and process changes to effect improvements in quality. These phases of the work are closely allied with that of the head inspector and may at times be combined with it.

Trouble shooting, in many cases, results from the inclusion within the grade standards of properties having too narrow a margin between conflicting properties. Research may often be needed to use different combinations of effects to widen the margin between conflicting properties to minimize cases of chronic trouble shooting. Frequently we also find in attempting to get the various quality properties to a higher level of quality that they do not respond to any one of many different effects attempted but instead respond to the same effect if used in combination with one or more other effects. A case in point is the fact that very seldom is resistance to penetration effects developed greatly as the result of application of surface effects, but they do respond to the same surface effects when accompanied by other changed effects such as increased hydration, increased finish, or in some cases, slacker sizing or differing places or amounts of application of the surface effects. Chasing down all the combinations made possible by geometric progression of such effects presents a control research program of considerable magnitude.

Additionally, control research also involves continuous research in the relationship between all measurable quality properties of paper and the specific use requirements of various printing and converting operations. Many times with comparatively slight changes a grade can be modified to be able to take in another use requirement thereby making the grade more versatile. This assists in the standardization of grades with the effect that the requirements of many more customers can be covered with a standard grade thereby avoiding extra changes. It also tends in many cases toward elimination of complaints resulting from using the grade for use requirement not originally contemplated. This sometimes results in making available extra sales possibilities.

Considerable time must be spent also in the development or improvement of control tests to make more measurable some of the specific properties demanded by the use requirements for which the conventional tests do not adequately serve. It is only after we have developed adequate sure-fire control tests for specific use requirements that will not betray us that we can safely attempt quality improvements or cost reductions. Frequently, in the absence of adequate control tests for specific use requirements, we attempt general conventional quality improvements that backfire on us with respect to one critical use requirement property.

Often also in our search for quality improvement for a specific use requirement we find it already present to a degree on a totally unrelated grade where we do not need it merely because of a fortuitous set of circumstances enclosed within its manufacturing standards. By thinking back to the principles of the unrelated grade, we frequently get suggestions for methods of improvement that we can inculcate into the grade that needs it. To illustrate this effect, a complaint received on a bristol grade because it will not allow the ink to dry, may lead us by consideration of the principles involved to a solution of a problem of resisting gloss ink penetration of a butter carton grade.

The control research function is distinctly an improvement function for which some sort of organizational provision is a practical necessity for its successful accomplishment.

Further in order to build systems of control for the various levels of organization and make provision for the improvement functions of quality improvement and cost reduction, organization must also make provision

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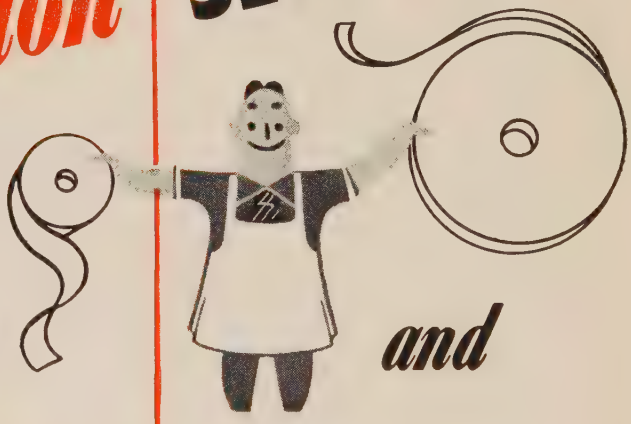

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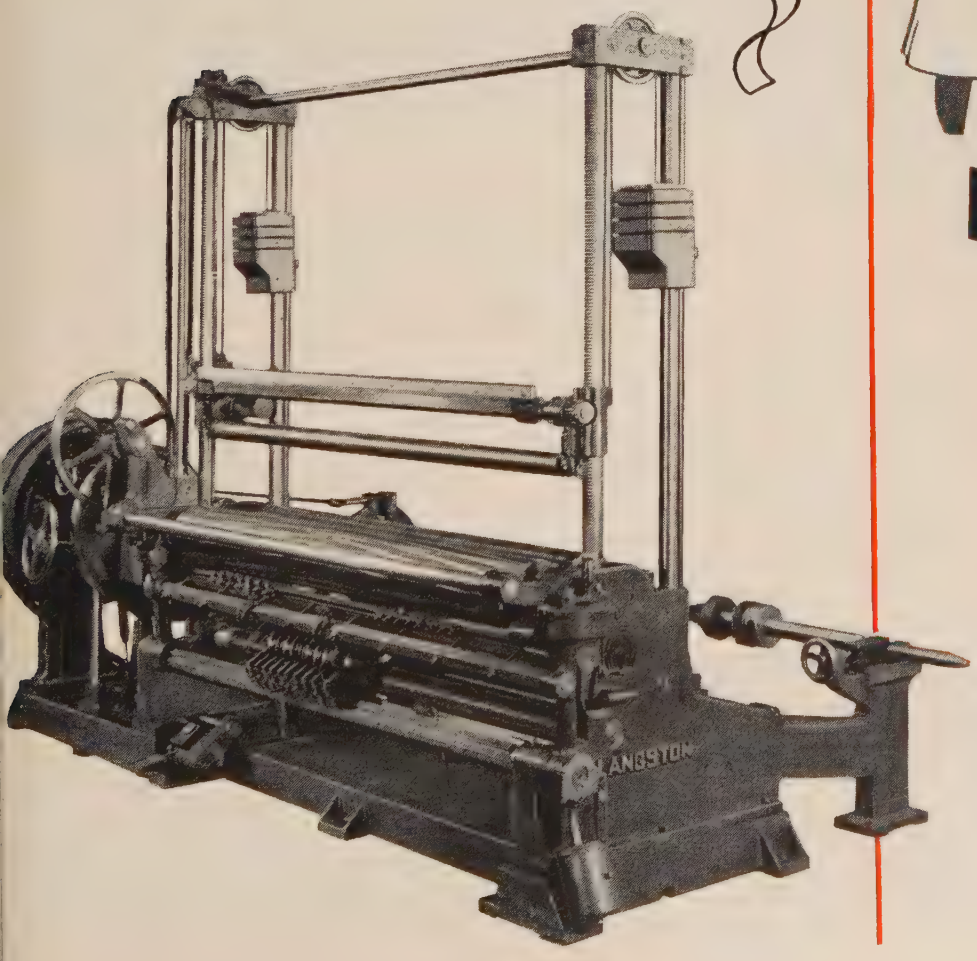

for the staff or improvement function as well as the line or routine function. All supervisory jobs bearing any part of the delegated production responsibility from administrative management through general management, production management, superintendents, and foremen possess two ever-present phases in common: the routine phase and the improvement phase. Organization must make provision for these two phases to be fully effective. In many cases at various organization levels, the two phases are merged in the activities of one person if the capacity in terms of time available and technical ability of a single person permits. The first staff or improvement function job of some one in the organization becomes that of devising an adequate control system which will not only control current routine quality but will supply an organizational mechanism for continuous quality improvement. Whether or not the improvement or staff function of building a quality control system is formally recognized by organizational provision, the function nevertheless exists. In the absence of formal staff organization the function must then be handled by the supervisor during what time he can spare from his strictly routine duties.

Obviously the basic necessity for quality control is a testing station at the end of the machine where all paper can be tested for its various physical properties by a tester, and the results recorded on a suitably designed quality log. The design of the quality log must be such that it emphasizes the most important elements on it. It must also be so designed that the relationship between various elements resulting from cause and effect can be easily traced. Many times the effectiveness of control is enhanced considerably by the proper design of the form.

Too much emphasis from the standpoint of control cannot be placed on the proper design of forms. It may even be advisable to repeat an element already provided for on another form such as putting freeness and furnish changes on the quality log in order to bring about the proper means of correlation of cause and effect. Forms should be so designed that they do all the thinking, reminding, and prompting for the operator. Forms when properly designed are more than a system for recording measurements. They are a learning method for the designer and a teaching method to the operator using them. Careful consideration should be given to what further can be accomplished along the lines of prompting, learning, and teaching in the redesign of subsequent printings of any form. Good forms are like good writing—they are the final product of many revisions. If we do not make the information easy to get by designing forms so they practically talk to us, we are opening the way for someone to follow the path of least resistance by not digging for the needed information, but instead, improvising along without it. After all, the paper industry which makes its living off forms should be one of the first to make most liberal use of them.

From quality and beater logs are listed to recap cards the essential machine settings, average, maximum, and minimum tests, and all furnish ingredients. These separate recap cards for furnish, machine and beater settings, and quality of each grade and weight show the complete history of the grade, extending back years in some cases. They show alternate furnishes and machine-setting conditions under which the grade has been successfully run and which can be used in case of temporary shortages of standard materials. They are also valuable for tracing the effect of variables quickly by comparison with test results in the case of complaints. From these also, the standard furnish and quality tests and standard speeds can be quickly chosen for inclusion on the standard run instruction card. Separate complaint history cards list all the complaints of the grade, extending back for years. Similarly a history of all special-furnish trials together with tests are listed on a separate trial card. These cards together with several last-run samples and last color match are then assembled into a run package in a large envelope so that when the run occurs, a complete history of furnishes, physical tests, samples, machine and beater settings, complaint and trial history, as well as color matches, are available for the use of tour boss, beater engineer, or superintendent for quick reference in case of any question arising during the run.

In order to control specifically, it is also necessary to take many different types of samples. The tester takes two full-machine width strips about 3 feet across, one of which he cuts off for the use of the machine tender and the other which he uses for testing purposes. The back tenders take regular samples in 12 by 12-inch size, from front, middle, and back, which they weigh and caliper for their own use in controlling between the testers sampling of the machine. The front and back samples are taken at the extreme edge of the deckle of the sheet in order to have a permanent record of the extreme edges for finish and caliper. These samples are packaged and filed by the head inspector, as representative samples throughout the run for examination or testing in the event of complaints. At the very start of the run a 12 by 12-inch sample is taken and signed by the tour boss to show the quality considered by him as passable for saving after the change. A similar "off" sample is also taken to indicate the last part of the run so that no part of the change is included in the previous run. Outturn samples in quantity are taken as representing the point at which the tour boss has ceased working on the run to indicate the general quality of the run in all respects. These samples are used for sales outturns and also are packaged for run library samples for comparison of various runs and for use as future potential standard samples. The latter are selected by comparison with the existing sample when the supply of standards is exhausted. Standard samples are printed up with grade, caliper, and customer designation. Package samples are also kept in the laboratory file of all "before and after" effects on trials of various sorts for purposes of research in the improvement of various qualities. Occasionally, also, on special runs samples are taken off machine rolls in full roll width to be available for testing bonding in the event of complaints, in cases where this feature is of a critical nature. On orders requiring critical width of rewound rolls, full width rewinder roll samples are also saved.

In order to identify samples positively, all samples are time punched with date and time. This procedure is most helpful in definitely establishing cause and effect relationships.

It is not too long ago in our industry that most quality was controlled almost entirely by observation and judgment with a bare minimum through crude testing. Through the use of improved testing methods we have

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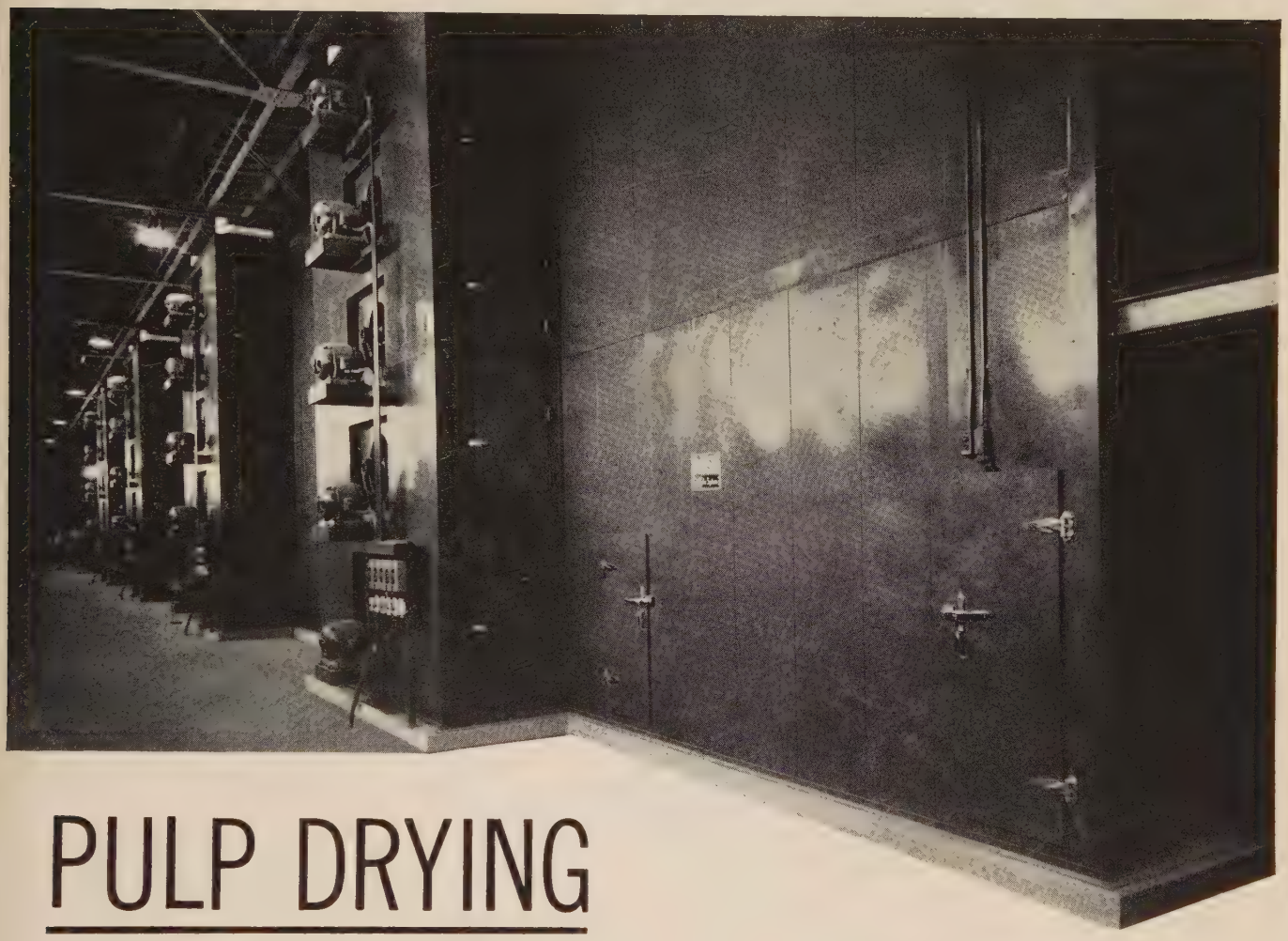

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been enabled to substitute testing, to a large but not yet fully complete degree, for observation and judgment. To do this, for our measurable use requirements, we all have had to spend considerable time in devising practical tests for control purposes that will give results that are in general agreement with our operating observations, judgment, and experience. When we can obtain such general agreement we can then accept the test as being more reliable and selective than the constantly shifting product of our eyes and judgment. The use of the brightness tester to measure the degradation of brightness of a crude waxing test offers an illustration of this principle. The development of such control tests have enabled us to measure, record, and correlate the results produced by material and process variables. Such control tests have done much to improve our general quality level continuously.

Even, however, at our relatively advanced status of today with respect to our standard testing methods we must nevertheless face the fact that for many practical considerations of our use requirements of paper, neither testing nor observation or judgment are complete of themselves. The one complements or supplements the other. We certainly have derived many benefits from testing but we still cannot get along entirely without observation and judgment of a high degree applied to our quality control. Despite our constant efforts to allocate more and more of our control to testing we still must make organizational provision for controlling at least part of our quality through the observation and judgment of people.

The first step in control through the observation and judgment of people is to organize for it by assigning the responsibility for control of specific defects and qualities to various members of the machine crew. Obviously the best and only effective place to separate out any defective paper is naturally at the paper machine as it is made. To make this effective all defective paper detected in the head inspector's subsequent inspection or in the various finishing operations must be considered as an inspection failure. Inspection failures must be treated more seriously than the making of defective paper to ensure adequate inspection at the machines. In organizing for quality control the question naturally arises whether the machine operators should be responsible for the inspection of qualities and defects, aside from physical testing, involving observation and judgment, or whether the tester should be responsible for all inspection as well as testing. It is our considered judgment that the machine operators should be responsible for their own quality inspection, aside from making physical tests. Testers in many cases may be too literal and, in general, have not had the extensive experience of machine men. Also machine operators will watch the general quality more minutely in order to establish a sensible balance between production speed and quality if they have responsibility for both elements. An inspector-tester is frequently not in a position to see all the defects and without machine experience might not recognize some of them if he did see them.

In organizing for machine inspection of observation and judgment qualities it is first necessary to compile a complete qualities and defect list from complaints, nonstandard paper lists, manufacturing instructions, and specifications. Inspection responsibilities for nonmeasurable qualities must be delegated among the various members of the machine crew and beater engineer, not so much with regard to their respective responsibilities for making the paper as with respect to the responsible individual's normal production duties placing him in a position to observe the defect. As a matter of fact, the inspection responsibilities of an individual may be completely at variance with his production responsibilities. Instead then of having several layers of increasing supervisory responsibility for all qualities and defects, we charge definite individual jobs with the specific inspection responsibility for various qualities and defects.

Essentially, the tour boss has supervisory responsibility for everything. He also has specific responsibility for making the change and okaying the "on and off" samples at the starting and ending of the run. He must inspect the full deckle strip at the start of the run. He is also specifically responsible for any defect showing on any samples extending for more than 2 hours. He must also personally check all the details of sizes, diameters, cores, etc., appearing on the orders. Essentially the tour boss makes all changes and gets the qualities right as indicated by the quality of the outturn samples. The machine tender then has the responsibility of holding this quality until he notifies the tour boss that the quality has changed. The tour boss is also responsible for initiating trouble-shooting procedures for under-standard quality as the necessity is reflected by the log tests or samples.

The machine tender is responsible for all defects common to all the paper showing up on the full deckle strip and the qualities indicated on the logs. Essentially, he has responsibility for a list of qualities and defects including weight, moisture, liner points, among others. These comprise all except those originating in the beater room and the intermittent defects. The machine tender also tests the paper for blows and loose liner personally.

The back tender removes from production any blows and loose liner on signal from the wet-end man. The back tender also has the responsibility for the detection and removal from production of all semi-intermittent defects visible from the calender stacks, including wet paper and off caliper. He regulates the caliper between reels by manipulating the stack weights and has specific responsibility for all mill work such as sizes, core diameters, etc.

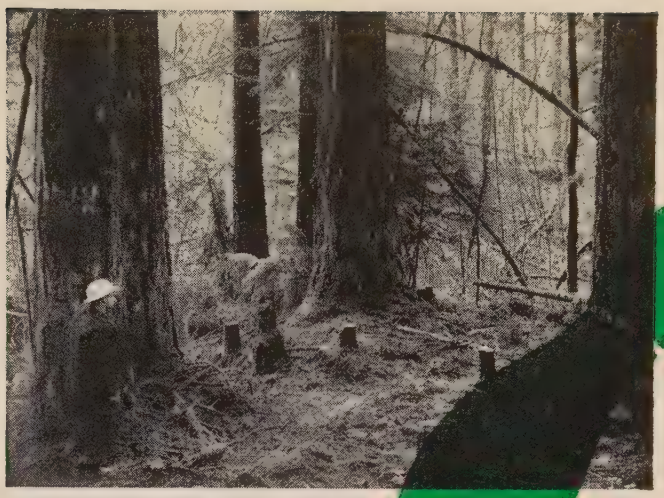
The wet-end man must detect all defects visible at the smoothing press and warn the back tender by signal of the presence of blows, checks, felt marks, skips, crush, etc.

The head cutterman has the complete responsibility for the removal of intermittent defects on sheets such as slime holes, dirt, streaks, shavings in the loads, etc. He is responsible for splices and proper roll marking.

The beater engineer has the responsibility for okaying the starting sample for color and watching the log and samples for color variation, sizing, and waxing color defects.

In order to establish supervisory control over the machine tenders and tour boss with respect to judgment factors, a procedure is instituted calling for the earmarking as "held for inspection" any paper below

Two Steps TOWARD WHOLE TIMBER CROP UTILIZATION



PRE-LOGGING which harvests small trees for use before they are damaged by the big log harvest.

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Pre-logging and re-logging are two Weyerhaeuser forestry practices that produce wood now useable in both the Weyerhaeuser sulphite and sulphate pulp mills. Logs as small as 4″ in diameter and 8′ in length are now handled by a recently developed small log hydraulic stream barker.

This means that the two sulphite pulp mills are producing pulp from logs of all sizes . . . the smaller ones supplied by pre-logging, which utilizes small trees that would otherwise be completely destroyed and wasted when larger trees are felled. The new sulphate pulp mill located adjacent to the lumber mill at Longview, produces its bleached kraft pulp from pre-logging operations, edgings and slabs from the Douglas Fir Saw-Mill . . . also from re-logging operations which follow the harvesting of the mature timber.

These advanced logging and whole crop utilization practices have made possible the economical operation of the Weyerhaeuser mills and greatly increased the pulp production from a given stand of timber.

standard however slightly in any respect, whether resulting from testing or inspection qualities. Such paper, naturally, is not processed by the finishing room until inspected by the superintendent. If the defect consists of an inconsequential imperfection, rather than a defect as such, it may be passed as "nonstandard paper," otherwise it is culled as broke. Any such nonstandard paper shipped is again earmarked by maintaining skid numbers or other means of identification to ensure locating it immediately in the customer's plant in case trouble should develop. Such fine distinctions between actual defects and slight imperfections of course call for careful judgment in order to give effect to the policy of never shipping a customer unsuitable merchandise. What may be only an imperfection for one specific use requirement may well be a defect for another use requirement, and vice versa. A basic principle to observe here in making such a fine distinction is the one of being certain of never risking the possibility of cheating a customer on any of his essential use requirements. A record of nonstandard paper shipped by grades as well as any type of imperfections then becomes the basic source of research problems for the elimination of even slight imperfections.

The final step in the quality control routine is the inspection of all records and samples gathered during the previous day by the head inspector for any defects that may have escaped the machine inspection. At this stage also, an examination is made for even any slight imperfections which may eventually result in defects if not noticed and studied at first sign of appearance. Any defective or nonstandard paper disclosed by this inspection is regarded as an inspection failure. This is listed by type of defect and individual responsibility for inspection of this defect for such corrective treatment as may be necessary. The head inspector posts the nonstandard paper from the broke log together with the inspection failures disclosed by his examination on a "held for inspection" list with samples for final disposition by the superintendent. Someone has well stated, "We cannot inspect quality into the product, it must be attained through control of the manufacturing process in all its elements." While the object of our inspection system routine is primarily to warn us that the manufacturing process is not under control, it also must have other functions if carried through to a degree of completion which will lead to real quality control through the process becoming under control.

From analysis of the nonstandard paper and broke reports methods of adaptation of various mechanical controls such as weight, consistency, and moisture regulators, liquid level gages, temperature controls, etc., will naturally be suggested and purchased or developed as one means of getting the process under control.

Principally, however, papermaking being an art, our most important result from our control system will be to give us tools with which to train our people. Training our people consists on the one hand of developing standard procedures or standard practice that represents the sum of the organization's combined knowledge of the particular subject at the minute. Such procedures should preferably be in written form, for important procedures at least, so that no doubt exists regarding what is the standard procedure. Deviations therefrom then resolve themselves into a clear-cut case of disobeying orders. In no case should a written procedure be allowed to fall into disuse. If the procedure is not correct, a new one should be issued.

More important than any type of disciplinary action, however, in getting people to do things right is the idea of helping them to do it right. In most cases proper attention to the teaching function will do much to practically preclude the necessity for ever thinking in disciplinary terms.

Are our instructions clear cut and complete? Do they answer the questions clearly of who, what, where, when, how, and why? Has our control system plenty of signposts, such as proper standards for furnish by types and quantities, standards for all significant quality properties, standard and last run samples, performance standards, proper machine settings standards, and also, proper standard procedures? Do we have available standard procedures for trouble shooting of various kinds outlining the steps to take in consecutive logical order from simplest to most drastic for elimination of common defects and improvement of measurable qualities? Some of the more drastic steps in a trouble shooting procedure might be as drastic as cutting off the felt, changing the wire, or shutting down the machine. Do we have adequate control testing methods, or at least empirical ones for difficultly measurable properties? Have we easily available all accumulated experience such as complaint summaries, experimental trial tests, and warnings of past mistakes and mishaps, so that they will literally "prompt" the operator to the correct action, even when he may be following the path of least resistance, which we all do at times? Above all, when a mistake is made by one of our better operators do we possess sufficient grace to consider it might be our fault in not setting up sufficiently adequate signposts to have prevented the mistake? Will we attempt to institute another signpost to replace the one that failed us? It is only when we have fulfilled all of the above conditions and have clearly bent over backward in blaming ourselves that we would be justified in attempting disciplinary measures.

In helping our operators do we spend sufficient time in teaching the why of our orders? People, generally, are found to obey orders much more gracefully when they know why they are instructed to follow an elaborate procedure. We even find that most people will subject themselves willingly to inconvenience and even discomfort if they are convinced that the necessities of the work situation demand this from them rather than its being caused by the arbitrary whims of a supervisor. Getting people under control involves, in many cases, a radical change in deepseated habits of long standing. To change such bad habits is a long process and is only arrived at by maintaining great patience and perseverance. In many cases to change long time careless habits to new more careful ones calls for repeated teaching through meetings procedures, signposts, and all the other devices at our command. Fortunately, however, it later becomes almost equally difficult to break the new more careful habits once they have been ingrained as habits into the routine. As a matter of fact, newcomers almost invariably adopt the pace of the organization with respect to work habits. If the general tone of working habits is good, they quickly develop good ones

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CHEMICALS FOR INDUSTRY

ROHM & HAAS COMPANY

THE RESINOUS PRODUCTS DIVISION

Washington Square Philadelphia 5, Pa.

The Resinous Products Division was formerly The Resinous Products & Chemical Company

themselves, and vice versa. This has particular application to the training of our people for their responsibility in watching over the elements of quality control calling for observation and judgment factors. One medium for teaching used in this phase of control consists of a form which is sent to the tour boss of each shift daily, listing by individual responsibility the inspection failures of his shift together with samples of the defects as disclosed by the head inspector's examination of the samples and logs.

From a classification of our nonstandard paper we can observe, at times, that certain of the defects seem to be chronic. The fact that they are chronic often indicates that the specific property does not have a sufficiently great safety margin as the result of the materials, equipment, or procedures in standard use. Naturally also, such defects do not always respond readily to the standard trouble shooting procedure for them. In such a case this indicates a need for research on the property, and its control becomes largely a staff function and as such should be removed from the operator's responsibility for control.

One phase of this subject which I feel sure is thoroughly familiar to all of us is that of customer complaints. Here we have discussed at great length a policy of, above all, never irritating our customers with defective paper and how to develop a control system that will preclude them, and yet we all do, nevertheless, get complaints from our customers.

All complaints should be treated formally by numbering them, making several copies, and listing them on a large chart in the machine room where they will serve as reminders. Copies of the letters should be posted in the machine room as well as being kept in a production office file.

Complaints should also be analyzed very carefully by recourse to the run reference samples (F-M-B) throughout the run which are maintained for this purpose. Machine and beater logs and quality and furnish recap cards are also valuable in analyzing the underlying fundamental cause of the complaint.

A complaint of the inspection failure type is due to a breakdown or a loophole in the quality control system which must be either rebuilt or plugged up before the system can again be effective. Unless extreme negligence of any part of the inspection system is clearly evident, it is best to assume that the system is not adequate and needs amplification. To this end there should be instituted into the system a reminder, in the form of a routine procedure, which, if it is carried out, will automatically preclude recurrence. If, for example, a complaint on off color or light weight occurs, which the log clearly shows came from part of the run change immediately ahead or behind it, the procedure of taking "on" and "off" samples can be instituted. Such a complaint actually was the forerunner of this particular phase of our routine procedure and no further complaint of this nature has been received for 10 years. A procedure, for example, to eliminate complaints from plates breaking as the result of trim strips getting caught in the cutter boxes can be instituted to allow them to roll up on the floor or to arrange a mechanism to blow them away by air. By resorting to specifically designed formalized procedures of this sort we can expect with assurance that the complaint source will be eliminated. In any event, it will be far easier to check on the routine performance of the procedure designed for its elimination than it will be to check on the effectiveness of its elimination without the procedure. Should a recurrence take place the control procedure instituted must be more carefully examined and modified or completely changed to another one which may be more effective. If we deliberately institute a specific procedure to eliminate each type of complaint arising from inspection failures, we should eventually get one to eliminate it completely. We must indeed work here on the principle that, "If at first you don't succeed, try again." If it is still possible for a reasonably careful and average operator to allow a serious inspection failure, then without a doubt the system is not effective enough and it should be improved.

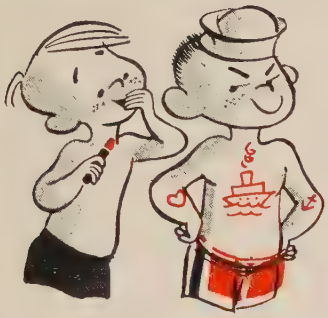
Great care must be taken never to blame operators unjustly in the case of complaints for mistakes resulting from their lack of knowledge or inadequate instructions. Scrupulous honesty of thinking must be used in reviewing the adequacy of instructions, standards, and standard procedures given the operators. Do they completely fulfill the requirements of adequate instructions in that they tell—who, what, where, when, why, and how? As a matter of fact, much more benefit will be derived from laying the stress completely on attempting to find out exactly what happened rather than where to attach blame. Aside from clear-cut cases of gross negligence, the blame for complaints other than those involving routine mill work, usually rests with management for not devising an adequate control system in all its ramifications of records, recaps, samples, standards, procedures, teaching, safety margins, research, signposts, and organization. The whole psychology of control is to teach our operators the importance of being responsible and to go faithfully through the quality control procedures designed to protect them and management alike in serving their common boss, the customer. In general, operators will in most cases respond to the environment that management produces and will take their responsibilities to the same degree and work about as hard as their supervisors do.

To sum up—effective quality control is the most important single control that we have in our tool kit of techniques because it essentially forms the beginning of a complete control system extending through production, cost, and budgetary controls.

One of the greatest difficulties confronting us, at lower organizational levels, in our efforts to achieve continuous quality improvement and cost reduction effects is the necessity for crossing strict organizational lines of responsibility and authority. The design of a complete control system therefore demands organizational provision of some sort for a clear-cut recognition of the staff or improvement function. Truly effective quality control together with its almost intertwined neighbor, cost control, demands a welding together of many functions such as labor relations policy determination, routine quality control, operation, research, and cost accounting. This necessity makes the development of a complete control system the responsibility of the lowest level of management that has organizational control of all these functions.

Received Dec. 8, 1950. Presented at a meeting of the New England Section of the Technical Association of the Pulp and Paper Industry, Holyoke, Mass., Nov. 17, 1950.

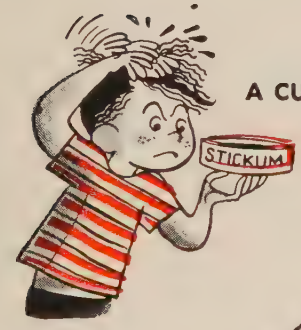
What "TYPE" Should Your Paper Be?



A GOOD PRINTER?

A WATER RESISTER?

AN EASY FOLDER?



A CURL REDUCER?

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S/V CEREMULS help give these characteristics—at low cost

Whatever type of paper you make, you can make it better—and at lower cost—with the help of these famous Socony-Vacuum wax emulsions.

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TAPPI Fibrary

This catalog is the first to be published for the TAPPI Fibrary, for which collection efforts were initiated in October, 1949. The task has just begun; many samples are in process of collection and there are many fibers which are needed. It is hoped that contributions of missing species will be forthcoming from TAPPI members so this fiber collection may continue to enlarge and increase in value. Addenda to this index will be published as occasion requires.

A representative specimen of each fiber may be obtained from the Fibrary, The Institute of Paper Chemistry, Appleton, Wis. Each sample has a number which may be used in ordering. A service charge of 25 cents ($0.25) per sample has been established to help defray the cost of handling, packaging, and postage. Remittance of the necessary fee at the time of ordering will help to keep these costs as low as possible.

To date the custodians of the Fibrary have not checked the fibers submitted, as to species and process. It is hoped that, in those cases where this is possible, they will be able to do so before shipping any samples.

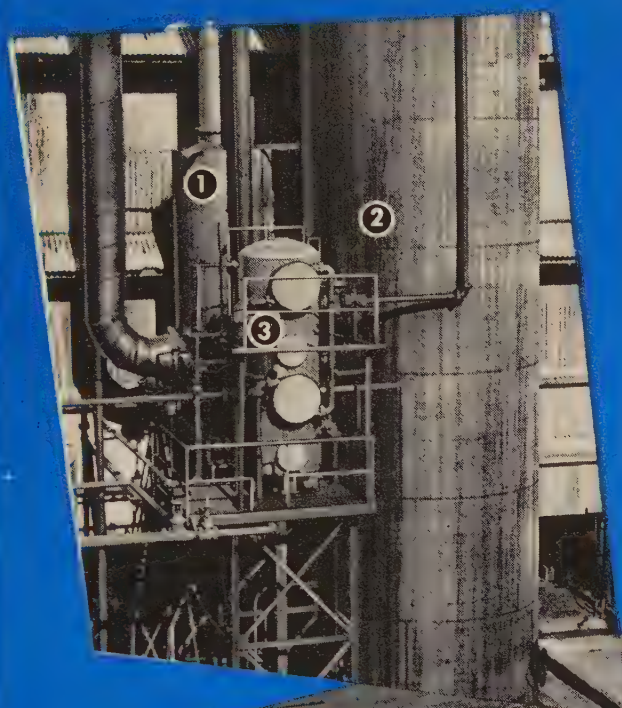
The amount of pulping data given by the donor varies considerably in different samples. Because of space limitations many of the details which have been sent with the sample will not be published but will be filed with the sample for reference purposes. Naturally, it is desirable to have complete details, if available.

No acknowledgment has been made in the catalog to the donors for the various samples. In many instances several companies or individuals offered the same fibers and while duplication is inevitable, every effort was made to avoid it to conserve space without decreasing the effectiveness of the fiber collection. Unquestionably, the gratitude of the industry is due these anonymous donors.

	Species	Location grown	Process	Degree of cooking	Bleaching	Remarks
1.	Ambari hemp (*Hibiscus cannabinus*)	India	Soda	Soft	None	...
2.	*Arundo donax* reed	Texas	Kraft	Soft	None	...
3.	Aspen (*Populus tremuloides*)	Wisconsin and Michigan	Groundwood	...	None	...
4.	Aspen (*Populus tremuloides*)	Minnesota	Groundwood	...	Full	Peroxide bleached
5.	Aspen (*Populous tremuloides*)	Wisconsin and Michigan	Neutral sulphite	...	None	Bauer refiner
6.	Aspen (*Populus tremuloides*)	Wisconsin and Michigan	Neutral sulphite	...	Semi	Same pulp as previous; single stage calcium hypochlorite bleach
7.	Aspen (*Populus tremuloides*)	Minnesota	Neutral sulphite	...	None	Asplund defibrator; sodium sulphite and caustic soda
8.	Aspen (*Populus tremuloides*)	Minnesota	Neutral sulphite	...	None	Given additional refining over previous sample
9.	Aspen (*Populus tremuloides*)	Minnesota	Kraft	Soft	None	...
10.	Aspen (*Populus tremuloides*)	Minnesota	Kraft	Soft	Full	...
11.	Balm-of-Gilead (*Populus balsamifera*)	Ontario	Kraft	Soft	None	...
12.	Barley straw (*Hordeum vulgare*)	Holland	Alkaline sulphite	Soft	None	Sodium sulphite + caustic soda
13.	Beech, European (*Fagus sylvatica*)	France	Acid semichemical	...	None	AB1 process
14.	Beech, European (*Fagus sylvatica*)	France	Acid semichemical	...	Full	AB1 process
15.	Beech, European (*Fagus sylvatica*)	Yugoslavia	Sulphite	20° Sieber	Full	...
16.	Beech, European (*Fagus sylvatica*)	Yugoslavia	Sulphite	20° Sieber	Full	Mobilized
17.	Birch, European white (*Betula alba*)	Sweden	Kraft	Medium	Full	...
18.	Birch, Swedish (*Betula verrucosa* and *pubescens*)	Sweden	Kraft	Medium	Full	Final stage bleach with chlorine dioxide
19.	Cantala fiber (*Agave cantala*)	Java	Soda	Soft	None	...
20.	Caroa fiber (*Neoglaziovia variegata*)	Brazil	Soda	Soft	None	...
21.	Chestnut, Spanish (*Castanea sativa*)	France	Soda	Medium	Full	...
22.	Chestnut, Spanish (*Castanea sativa*)	Italy	Kraft	3.0 Roe	None	Extracted chips
23.	Cotton seed hair (*Gossypium hirsutum*)	United States	Soda	...	...	Nitration type
24.	Cotton seed hair (*Gossypium hirsutum*)	United States	Soda	...	...	Acetate plastics
25.	Cotton seed hair (*Gossypium hirsutum*)	United States	Soda	...	...	Viscose
26.	Cotton seed hair (*Gossypium hirsutum*)	United States	Soda	...	...	Writing papers, etc.
27.	Cotton seed hair (*Gossypium hirsutum*)	United States	Soda	...	...	Viscose, sheet
28.	Cotton seed hair (*Gossypium hirsutum*)	United States	Soda	...	...	Cellulose ethers
29.	Cotton seed hair (*Gossypium hirsutum*)	United States	Soda	Soft	Full	Second cut and mill run linters
30.	Cotton seed hair (*Gossypium hirsutum*)	United States	Soda	Soft	Full	Unbleached muslin cuttings
31.	Cotton stalk (*Gossypium hirsutum*)	United States	...	...	None	...
32.	Cotton stalk (*Gossypium hirsutum*)	United States	...	...	Semi	...
33.	Douglas-fir (*Pseudotsuga taxifolia*)	Oregon	Asplund	...	None	Hardboard pulp
34.	Douglas-fir (*Pseudotsuga taxifolia*)	Oregon	Asplund	...	None	Bauer refined; hardboard pulp

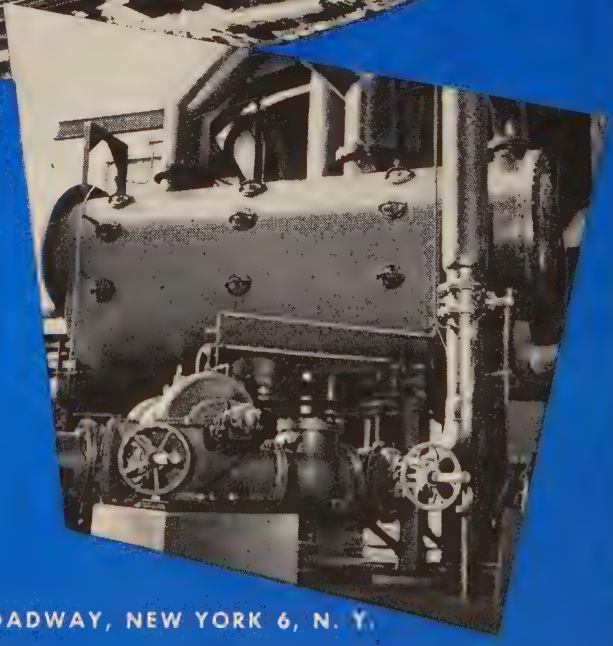
harnessing HEAT DOLLARS *at St. Regis*

with FOSTER WHEELER BLOWDOWN RECOVERY SYSTEMS



At the St. Regis Paper Mills in Pensacola, Florida, two types of blowdown recovery systems are in operation. In the Vapor-to-Liquid System *(left)* steam is condensed in the atmospheric condenser (1) and hot condensate is stored in the accumulator tank (2). This condensate, contaminated by some black liquor and pulp, is flashed under vacuum and the resultant clean vapors heat mill water in the tubes of the flash heater (3).

In the Surface Condenser *(below)* mill water flowing through the tubes is heated by steam condensing in the shell. The expansion chamber at vapor inlet prevents pulp from reaching tube bundles.



Properly engineered recovery equipment not only ***costs you less*** in the long run but ***increased operating efficiency*** and ***large fuel savings*** may also be enjoyed from the outset.

If you are contemplating improvements at your mill, our specialists on pulp and paper mill equipment will be glad to help you with your engineering problems.

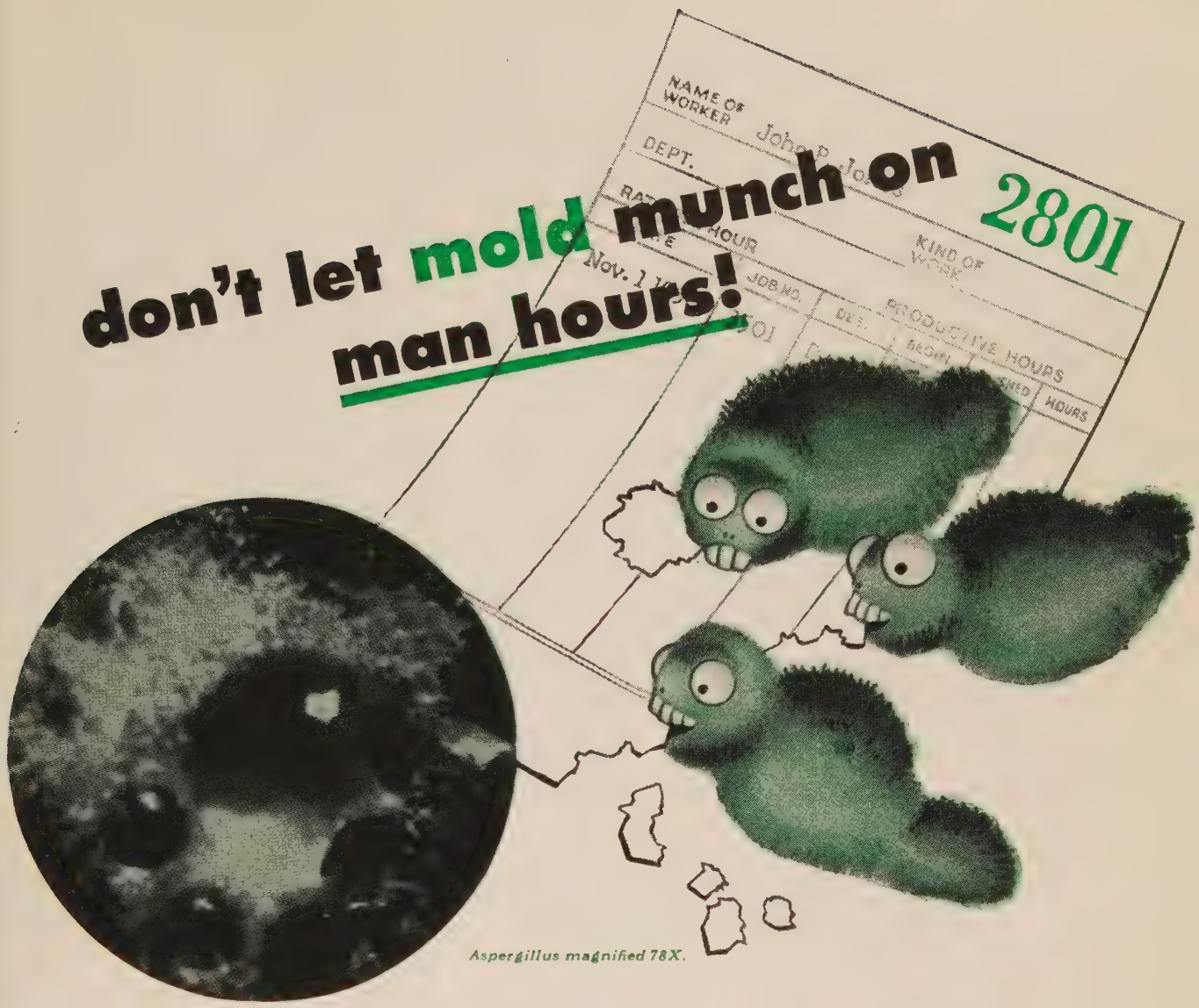
Foster Wheeler Blowdown Recovery Systems are discussed in the May, 1950 issue of *Heat Engineering*. Write for a copy:

FOSTER WHEELER CORPORATION • 165 BROADWAY, NEW YORK 6, N. Y.

FOSTER WHEELER

	Species	Location grown	Process	Degree of cooking	Bleaching	Remarks
35.	Douglas-fir (*Pseudotsuga taxifolia*)	Pacific Northwest	Soda	Soft	None	. . .
36.	Douglas-fir (*Pseudotsuga taxifolia*)	Oregon	Kraft	Soft	None	. .
37.	Douglas-fir (*Pseudotsuga taxifolia*)	Oregon	Kraft	Soft	Semi	. . .
38.	Douglas-fir (*Pseudotsuga taxifolia*)	Pacific Northwest	Kraft	Medium	None	. . .
39.	Douglas-fir (*Pseudotsuga taxifolia*)	Pacific Northwest	Sulphite	Semichemical	Full	. . .
40.	Douglas-fir (*Pseudotsuga taxifolia*)	Pacific Northwest	Sulphite	Hard	None	Ammonia base
41.	Elephant grass (or bango reed)	South Africa	. . .	. . .	. . .	. . .
42.	Esparto (*Stipa tenacissima*)	Algeria	Soda	Medium	None	. . .
43.	Esparto (*Stipa tenacissima*)	Algeria	Soda	Medium	Full	Sodium hypochlorite bleach
44.	*Eucalyptus globulus*	California	Asplund	. . .	. . .	Roofing felts
45.	*Eucalyptus regnans*	Tasmania	Groundwood	. . .	None	Mixture with 5% *E. gigantea* and 5% *E. obliqua*
46.	Fique fiber (*Fourcroya macrophylla*)	. . .	Soda	Soft	None	. . .
47.	Fir, balsam (*Abies balsamea*)	Quebec	Groundwood	. . .	None	. . .
48.	Fir, balsam (*Abies balsamea*)	Minnesota	Groundwood	. . .	Full	Peroxide bleached
49.	Fir, balsam (*Abies balsamea*)	Ontario	Sulphite	Medium	None	. . .
50.	Fir, balsam (*Abies balsamea*)	Ontario	Sulphite	Medium	Full	. . .
51.	Fir, balsam (*Abies balsamea*)	Wisconsin and Minnesota	Kraft	Medium	None	. .
52.	Fir, red (*Abies magnifica*)	California	Kraft	Soft	None	. . .
53.	Fir, silver (*Abies alba*)	Germany	Groundwood	. . .	. . .	. . .
54.	Fir, silver (*Abies alba*)	France	Neutral sulphite	. . .	None	Mixed with Norway spruce; sawmill slabs, including some bark
55.	Fir, western balsam (*Abies* sp.)	British Columbia	Sulphite	Soft	None	. . .
56.	Fir, western balsam (*Abies* sp.)	British Columbia	Sulphite	Soft	Semi	. . .
57.	Fir, western balsam (*Abies* sp.)	British Columbia	Sulphite	Soft	Full	. . .
58.	Fir, white (*Abies concolor*)	California	Kraft	Hard	None	. . .
59.	Flax (*Linum usitatissimum*)	Holland	Soda	Medium	Full	. . .
60.	Flax (*Linum usitatissimum*)	Minnesota	Kraft	Medium	None	. . .
61.	Flax shives	Minnesota	Sulphur-ammonia	. . .	None	. . .
62.	Hemlock, eastern (*Tsuga canadensis*)	Nova Scotia	Sulphite	Hard	None	. . .
63.	Hemlock, eastern (*Tsuga canadensis*)	Nova Scotia	Sulphite	Medium	None	. .
64.	Hemlock, western (*Tsuga heterophylla*)	Pacific Northwest	Sulphite	Medium	None	. . .
65.	Hemlock, western (*Tsuga heterophylla*)	British Columbia	Sulphite	Soft	None	Dissolving pulp
66.	Hemlock, western (*Tsuga heterophylla*)	British Columbia	Sulphite	Soft	Semi	Dissolving pulp
67.	Hemlock, western (*Tsuga heterophylla*	British Columbia	Sulphite	Soft	Full	Dissolving pulp
68.	Hemlock, western (*Tsuga heterophylla*)	Pacific Northwest	Sulphite	Soft	None	Ammonia base
69.	Hemlock, western (*Tsuga heterophylla*)	Pacific Northwest	Sulphite	Soft	None	Magnesium base
70.	Hemlock, western (*Tsuga heterophylla*)	Alaska	Kraft	Medium	None	
71.	Hemlock, western (*Tsuga heterophylla*)	Oregon	Kraft	Medium	None	
72.	Hemlock, western (*Tsuga heterophylla*)	Oregon	Kraft	Medium	Semi	. . .
73.	Hemp rope		Soda	Medium	Full	Cooked in India, source unknown
74.	Incense-cedar (*Libocedrus decurrens*)	California	Kraft	Soft	Semi	. .
75.	Kozo (*Broussonetia papyrifera*)	Japan	Soda	Hard	None	. .
76.	*Lespedeza sericea*	Alabama	Kraft	. . .	None	. . .
77.	*Lespedeza sericea*	Alabama	Kraft	. .	Full	Sodium hypochlorite bleach
78.	*Lespedeza sericea*	Alabama	Pomilio	. . .	Full	. . .
79.	Manila fiber (*Musa textilis*)	Philippine Islands	Soda	Soft	None	. .
80.	Manila fiber (*Musa textilis*)	Philippine Islands	Alkaline sulphite	Soft	None	. . .
81.	Mauritius hemp (*Fourcroya gigantea*)	Mauritius	Soda	Soft	None	. . .
82.	Mesquite (*Prosopis juliflora*)	Texas	Kraft	Medium	None	. . .
83.	Mora (*Mora excelsa*)	British Guiana	Kraft	. . .	None	Dye extracted before cook
84.	New Zealand flax (*Phormium tenax*)	New Zealand	Kraft	Hard	None	. . .
85.	New Zealand flax (*Phormium tenax*)	New Zealand	Neutral sulphite	Hard	None	Board grade
86.	New Zealand flax (*Phormium tenax*)	Azore Islands	Alkaline sulphite	Medium	None	. .
87.	Oak, English (*Quercus robur*)	France	Soda	Medium	Full	. .
88.	Oak, post (*Quercus stellata*)	Texas	Kraft	Soft	None	.
89.	Oat straw (*Avena sativa*)	Holland	Alkaline sulphite	Soft	None	.
90.	Pine, Corsican (*Pinus laricio* var. *calabrica*)	Italy	Kraft	. . .	None	. .
91.	Pine, jack (*Pinus banksiana*)	Minnesota	Kraft	Soft	None	. . .
92.	Pine, jack (*Pinus banksiana*)	Minnesota	Kraft	Soft	Semi	Two-stage bleach
93.	Pine, jack (*Pinus banksiana*)	Minnesota	Kraft	Soft	Full	Five-stage bleach
94.	Pine, jack (*Pinus banksiana*)	Wisconsin	Kraft	Medium	None	. .
95.	Pine, jack (*Pinus banksiana*)	Minnesota	Sulphite	Soft	None	Sapwood
96.	Pine, jack (*Pinus banksiana*)	Minnesota	Sulphite	Soft	None	Sapwood; sodium base
97.	Pine, Jeffrey (*Pinus jeffreyi*)	California	Kraft	Medium	None	. . .
98.	Pine, loblolly (*Pinus taeda*)	Louisiana and Arkansas	Kraft	Medium	None	.
99.	Pine, lodgepole (*Pinus contorta* var. *latifolia*)	Pacific Northwest	Sulphite	Medium	None	.
100.	Pine, lodgepole (*Pinus contorta* var. *latifolia*)	Pacific Northwest	Sulphite	Soft	None	.

don't let mold munch on man hours!



Aspergillus magnified 78X.

Mold and bacteria can cause significant loss of man hours and material in the pulp and paper industry.

Dowicides work effectively and economically to protect starches, casein, glue and other decomposable adhesives, sizes or coating materials from attack by mold and bacteria. They also prevent mildew on lap stock in storage and the deterioration of paper board cartons and other paper products which may be stored or used under humid conditions.

Water and wax-soluble Dowicides will give full protection to various types of soap wrappers.

If you are not already using Dowicides for all-around microbial protection in *your* mill, contact your nearest Dow sales office today, or write to Midland. Dow maintains complete laboratory facilities to help solve your problems.

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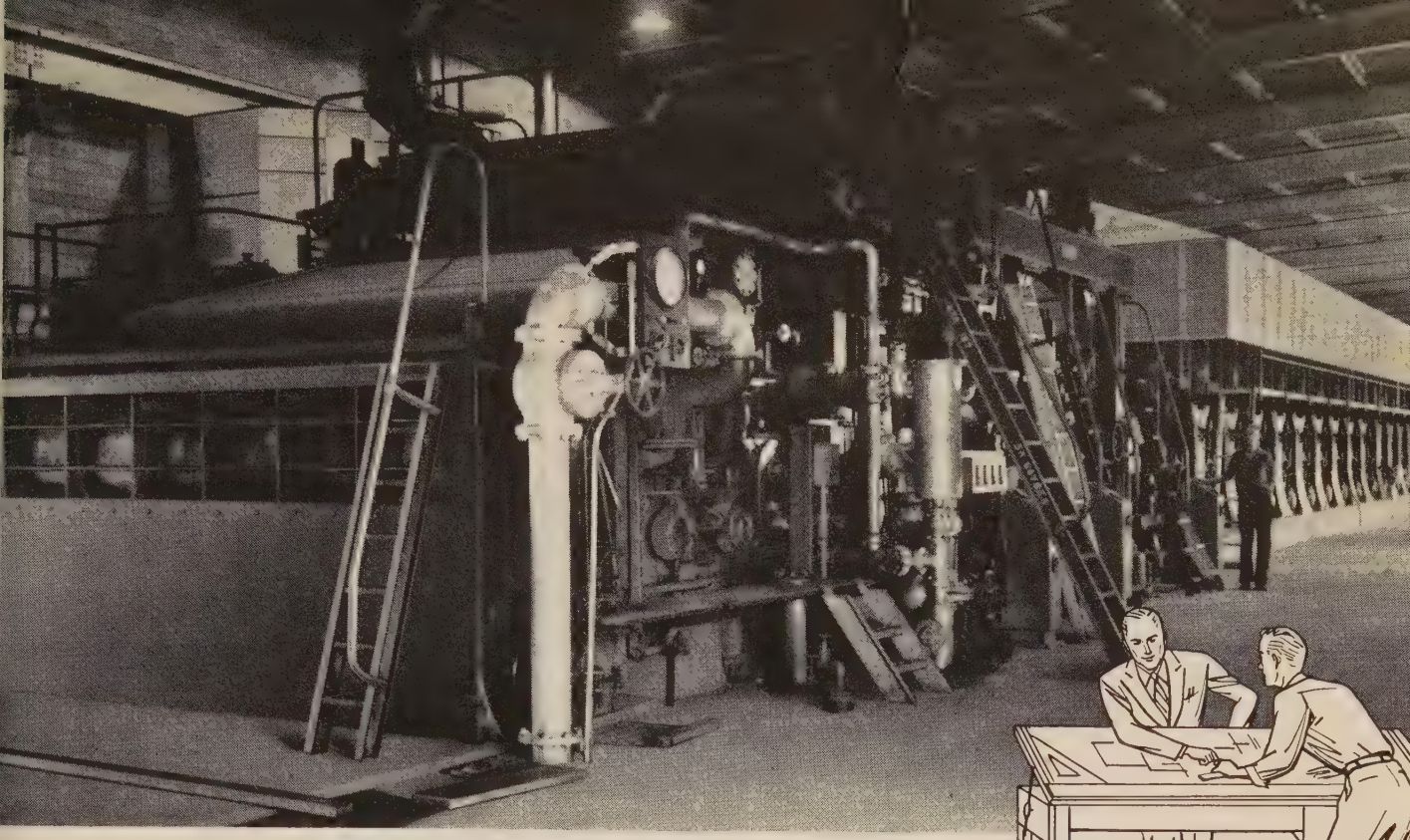
DOW

CHEMICALS

INDISPENSABLE TO INDUSTRY AND AGRICULTURE

	Species	Location grown	Process	Degree of cooking	Bleaching	Remarks
101.	Pine, lodgepole (*Pinus contorta* var. *latifolia*)	Pacific Northwest	Sulphite	Medium	None	Ammonia base
102.	Pine, lodgepole (*Pinus contorta* var. *latifolia*)	Pacific Northwest	Sulphite	Soft	None	Ammonia base
103.	Pine, lodgepole (*Pinus contorta* var. *latifolia*)	Pacific Northwest	Sulphite	Medium	None	Magnesium base
104.	Pine, lodgepole (*Pinus contorta* var. *latifolia*)	Pacific Northwest	Sulphite	Soft	None	Magnesium base
105.	Pine, lodgepole (*Pinus contorta* var. *latifolia*)	Montana	Kraft	Medium	None	...
106.	Pine, lodgepole (*Pinus contorta* var. *latifolia*)	California	Kraft	Medium	None	...
107.	Pine, longleaf (*Pinus palustris*)	Texas	Kraft	Medium	None	...
108.	Pine, maritime (*Pinus maritima*)	France	Sulphite	Soft	Full	...
109.	Pine, maritime (*Pinus pinaster*)	Italy	Kraft	...	None	...
110.	Pine, maritime (*Pinus pinaster*)	Italy	Kraft	...	None	...
111.	Pine, Mexican (*Pinus montzumae*)	Mexico	Kraft	Medium	None	...
112.	Pine, Monterey (*Pinus radiata*)	New Zealand	Kraft	Hard	None	Slabwood
113.	Pine, Monterey (*Pinus radiata*)	New Zealand	Kraft	Medium	None	Slabwood
114.	Pine, Monterey (*Pinus radiata*)	New Zealand	Kraft	Soft	None	Slabwood
115.	Pine, Monterey (*Pinus radiata*)	California	Asplund	Hard	None	Roofing felts
116.	Pine, mountain (*Pinus montana*)	Italy	Kraft	...	None	...
117.	Pine, Parana (*Araucaria brasiliana*)	Brazil	Sulphite	...	None	...
118.	Pine, Scotch (*Pinus silvestris*)	Sweden	Kraft	Hard	None	...
119.	Pine, Scotch (*Pinus silvestris*)	Sweden	Kraft	Medium	None	...
120.	Pine, Scotch (*Pinus silvestris*)	Sweden	Kraft	Medium	Full	...
121.	Pine, Scotch (*Pinus silvestris*)	Sweden	Kraft	Medium	Full	Bleached with chlorine dioxide
122.	Pine, Scotch (*Pinus silvestris*)	Finland	Kraft	7.7 Roe	None	...
123.	Pine, Scotch (*Pinus silvestris*)	Finland	Kraft	5.5 Roe	None	...
124.	Pine, Scotch (*Pinus silvestris*)	Finland	Kraft	2.6 Roe	None	...
125.	Pine, Scotch (*Pinus silvestris*)	Norway	Kraft	Soft	None	Includes 20% Norway spruce
126.	Pine, Scotch (*Pinus silvestris*)	Italy	Groundwood	...	None	Includes some spruce and poplar
127.	Pine, slash (*Pinus caribaea*)	Georgia	Kraft	Medium	None	...
128.	Pine, slash (*Pinus caribaea*)	Georgia	Kraft	Soft	None	...
129.	Pine, sugar (*Pinus lambertiana*)	California	Kraft	Medium	None	...
130.	Pine, Virginia (*Pinus virginiana*)	Maryland	Steamed and refined	...	None	Unpeeled wood; steamed in presence of water and hydrated lime, then Bauer refined
131.	Pine, white (*Pinus strobus*)	New York	Groundwood	...	None	Wall paper grade
132.	Pineapple (*Ananas comosus*)	Puerto Rico	Soda	...	...	...
133.	Pita floja (*Aechme magdalenae*)	Columbia	Soda	Medium	None	...
134.	Poplar (*Populus* sp.)	United States	Soda	...	None	...
135.	Poplar (*Populus* sp.)	United States	Soda	...	Full	One-stage bleach
136.	Poplar, Black (*Populus italica*)	Italy	Sulphite	30° Sieber	Full	Two-stage bleach
137.	Poplar (*Populus canadensis*)	Italy	Sulphite	20° Sieber	Full	Nobilized
138.	Poplar (*Populus canadensis*)	Italy	Sulphite	20° Sieber	Full	Heartwood
139.	Poplar, white (*Populus alba*)	Italy	Groundwood	...	None	...
140.	Poplar, white (*Populus alba*)	Italy	Kraft	3.0 Roe	None	...
141.	Poplar, white (*Populus alba*)	Italy	Soda	...	...	...
142.	Port Orford cedar (*Chamaecyparis lawsoniana*)	Oregon	Sulphite	Medium	None	...
143.	Port Orford cedar (*Chamaecyparis lawsoniana*)	Oregon	Kraft	Hard	None	...
144.	Ramie (*Boehmeria nivea* var. *tenacissima*)	Brazil	Kraft	Medium	None	...
145.	Red-cedar, western (*Thuja plicata*)	Pacific Northwest	Sulphite	Medium	None	...
146.	Red-cedar, western (*Thuja plicata*)	Pacific Northwest	Sulphite	Medium	None	Magnesium base
147.	Red-cedar, western (*Thuja plicata*)	Pacific Northwest	Sulphite	Soft	None	Ammonia base
148.	Rice straw (*Oryza sativa*)	Italy	Neutral sulphite	...	Semi	Sodium sulphite and caustic soda; single-stage bleach with calcium hypochlorite
149.	Rye straw (*secale cereale*)	Holland	Alkaline sulphite	Soft	None	Sodium sulphite and caustic soda
150.	Sabai grass (*Eulalipos binata*)	India	Soda	Medium	Full	...
151.	*Sansevieria metallica* fiber	Florida	Soda	Soft	None	...
152.	Sisal (*Agave sisalina*)	Africa	Soda	Soft	None	...
153.	Sorghum (*Sorghum vulgare*)	Italy	Neutral sulphite	...	None	Sodium sulphite and caustic soda
154.	Spruce, black (*Picea mariana*)	Minnesota	Sulphite	Soft	None	...
155.	Spruce, black (*Picea mariana*)	Minnesota	Sulphite	Soft	Full	...
156.	Spruce, black (*Picea mariana*)	Minnesota	Sulphite	Soft	None	Sodium base
157.	Spruce, black (*Picea mariana*)	Minnesota	Sulphite	Soft	Full	Sodium base
158.	Spruce, black (*Picea mariana*)	Ontario	Sulphite	Soft	None	Mitscherlich process
159.	Spruce, black (*Picea mariana*)	Ontario	Sulphite	Soft	Full	Mitscherlich process
160.	Spruce (black and/or white)	Wisconsin	Kraft	Medium	None	...
161.	Spruce (black and/or white)	Minnesota	Groundwood	...	Full	Peroxide bleach
162.	Spruce (black and/or white)	Quebec	Groundwood	...	None	...
163.	Spruce, eastern (*Picea* sp.)	New York	Groundwood	...	None	Presteamed; includes 25% balsam fir
164.	Spruce, Engelmann (*Picea engelmanni*)	Colorado	Sulphite	Medium	None	...



Puseyjones Cylinder Machine with a Stream-Flow Wet End—the most modern felt machine ever built.

Cylinder Machines
TAILORED TO YOUR NEEDS

If your 1951 plans call for a new Cylinder Machine or the rebuilding of an existing one, ask a Puseyjones engineer to show you this record:

Each *new* Puseyjones Cylinder Machine has broken former speed limitations *regardless of the product being made.*

Every Cylinder Machine *rebuilt* by Puseyjones, has reached and exceeded expectations—with better sheet quality, higher speed possibilities and furnish economies.

Outstanding Cylinder Machine improvements, like the Goldsmith Stream-Flow Vat System, are available on new or rebuilt machines. Machine change-overs are often accomplished in several steps – first, Stream-Flow Vat Systems with Flow Spreaders, later Vacuum Cylinder Molds, followed by Press improvements and added Dryer capacity.

Whatever your product may be—container board, folding boxboard, bristol and index, cylinder paper, felt, or special products – Puseyjones engineers will tailor the equipment to meet your needs. Write or call us today.

THE PUSEY AND JONES CORPORATION

Established 1848. Builders of Paper-Making Machinery

Fabricators and Welders of All Classes of Steel and Alloy Products

Wilmington 99, Delaware, U.S.A.

THE SIGN OF TRADE MARK SUPERIOR QUALITY

	Species	Location grown	Process	Degree of cooking	Bleaching	Remarks
165.	Spruce, Engelmann (*Picea engelmanni*)	New Mexico	Kraft	Soft	None	...
166.	Spruce, Norway (*Picea excelsa*)	Sweden	Sulphite	Hard	None	...
167.	Spruce, Norway (*Picea excelsa*)	Sweden	Sulphite	Soft	None	...
168.	Spruce, Norway (*Picea excelsa*)	Sweden	Sulphite	Medium	Full	...
169.	Spruce, red (*Picea rubens*)	Maine	Sulphite	Medium	None	Mixed ammonia and calcium base
170.	Spruce, Sitka (*Picea sitchensis*)	Alaska	Kraft	Soft	Full	...
171.	Sugar cane bagasse (*Saccharum officinarum*)	Louisiana	Milled for juice extraction	Raw	None	Variable content of leaves and stalk
172.	Sugar cane bagasse (*Saccharum officinarum*)	Louisiana	Steamed	Hard to medium	None	Disintegrated in water and steam digested for 1 hour
173.	Sugar cane bagasse (*Saccharum officinarum*)	Louisiana	Kraft	Hard	None	66% Yield
174.	Sugar cane bagasse (*Saccharum officinarum*)	Louisiana	Kraft	Soft	None	60% Yield
175.	Sugar cane bagasse (*Saccharum officinarum*)	Philippine Islands	Pomilio-Celdecor	...	Full	...
176.	Sunn (*Crotalaria juncea*)	India	Alkaline sulphite	Soft	None	...
177.	Sweetgum (*Liquidambar styraciflua*)	Arkansas	Kraft	Hard	None	...
178.	Sweetgum (*Liquidambar styraciflua*)	Arkansas	Kraft	Soft	None	...
179.	Sweetgum (*Liquidambar styraciflua*)	Louisiana	Kraft	Soft	None	...
180.	Tamarack (*Larix laricina*)	Minnesota	Kraft	Soft	None	...
181.	Tamarack (*Larix laricina*)	Minnesota	Sulphite	Medium	None	...
182.	Tupelo, black (*Nyssa sylvatica*)	Louisiana	Neutral sulphite	Semichemical	None	...
183.	Wheat straw (*Triticum vulgare*)	Indiana	Neutral sulphite	Hard	None	...
184.	Wheat straw (*Triticum vulgare*)	Illinois	Neutral sulphite	Soft	None	...
185.	Wheat straw (*Triticum vulgare*)	Illinois	Neutral sulphite	Soft	Full	...
186.	Wheat straw (*Triticum vulgare*)	Holland	Alkaline sulphite	Medium soft	None	Sodium sulphite and caustic soda
187.	Wheat straw (*Triticum vulgare*)	Illinois	Kraft	Soft	None	Hydrapulper
188.	Wheat straw (*Triticum vulgare*)	Illinois	Kraft	Soft	Full	Hydrapulper
189.	Wheat straw (*Triticum vulgare*)	Illinois	Soda	Soft	None	Hydrapulper
190.	Wheat straw (*Triticum vulgare*)	Illinois	Soda	Soft	Full	Hydrapulper
191.	Wheat straw (*Triticum vulgare*)	Illinois	Lime-soda	Soft	None	Washed and beaten
192.	Wheat straw (*Triticum vulgare*)	Indiana	Lime-soda	Hard	None	...
193.	Wheat straw and oat straw	Holland	Alkaline sulphite	Medium soft	None	50-50 mixture; sodium sulphite and caustic soda
194.	Yawa fiber (*Vigna sinensis* var. *textilis*)	Nigeria	Soda	Soft	None	...

WELDCO ROLLS

FOR LONGER SERVICE LIFE

LOWER OPERATING COSTS

They're Standard Equipment in America's Biggest Paper Mills

• And you'll find them in many textile, food, and chemical plants, too. Weldco Monel-covered rolls are carefully ground for accuracy and uniformity . . . then polished to a fine, smooth surface that reduces friction and keeps materials from sticking.

Weldco rolls are preferred, also, because of their high strength and stiffness — because they do not sag or "whip," do not corrode or accumulate pitch. They need very little cleaning, will withstand acids and alkaline white waters in fine shape, and show little wear on fourdrinier wires.

The Weldco covering process can be applied to both new and reclaimed rolls, eliminating the cost of new cores and providing substantial savings. Write or phone today for complete information and descriptive literature.

THE YOUNGSTOWN WELDING & ENGINEERING COMPANY

OAKWOOD AVENUE . . . YOUNGSTOWN 9, OHIO

NEW FREE RESEARCH KIT!

4 CHEMICAL DERIVATIVES from starch

The best place to keep abreast of new developments of new chemical derivatives from starch is right in your own experimental laboratory. Try applying the characteristics and potentialities of National's four basic chemical derivatives* from starch to your paper-making problems. Complete descriptions and known applications are included in the kit.

PICK UP YOUR KIT at National Starch Convention Headquarters, Hotel Commodore. We'll be happy to answer any of your questions: Frank Greenwall, Don Pascal, Jack Fitzgerald, Les Klempner, Fred Bradley, Dick Stevens, John Clay, Bill Stone, Ben Gordon, Harry Kaufmann, Dr. T. A. White, Frank Brundage, Leonard Wood, Harold Zahrndt.

National
STARCH PRODUCTS

270 Madison Avenue, New York 16, N. Y.

*Patented, or patent pending

Assures maintenance-free protection

Johns-Manville WEATHER-PROTECTED INSULATION

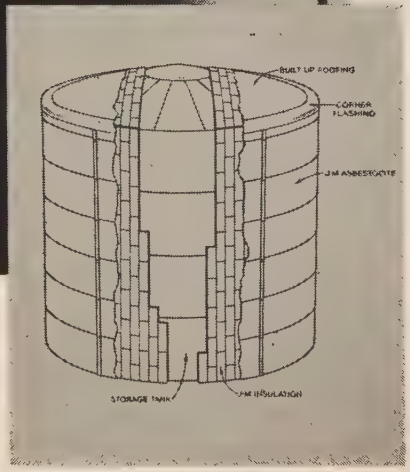
for tanks and vessels

If you have outdoor—or indoor—tanks and vessels . . . such as the multiple effect evaporators shown above . . . it will pay you to look into Johns-Manville Weather-Protected Insulation.

Weather-Protected Insulation pays because it does a twofold job: 1. It provides the close temperature control so important in the process industries; 2. It assures a maintenance-free insulation job.

Basically, this Johns-Manville Weather-Protected Insulation specification consists of standard J-M Insulations over which is applied Johns-Manville Asbestocite (a tough, strong asbestos-cement sheet) to protect the insulation from the weather or from wetting due to normal plant operations. Shielded in this manner, the insulation maintains its original efficiency and requires no periodic maintenance.

If you wish, a Johns-Manville Insulation Engineer will be glad to survey your equipment and make appropriate recommendations. For further details, send for a copy of folder IN-121A. Address Johns-Manville, Box 290, New York 16, N. Y.



Cutaway drawing shows how Johns-Manville Weather-Protected Insulation is applied to a tank—Standard methods for mechanical securement of the insulation are used. Asbestocite sheets are then applied over the insulation, following a simplified Johns-Manville specification.

JOHNS-MANVILLE JM PRODUCTS

Johns-Manville *first in* INSULATIONS

DU PONT PIGMENT COLORS

for

WALL PAPER
COATED PAPER
FLINTED PAPERS
BEATER DYEING

STANDARDIZED PIGMENTS to MEET the EXACTING REQUIREMENTS for PAPER COLORING

THE DU PONT LINE includes economical pigments of highest quality. There are colors for beater dyeing which have been especially processed for quick and thorough dispersion in water. They have better strength than the usual lakes for beater coloring. Use them for papers which must have good lightfastness.

All these Du Pont pigments are standardized for properties important to their use in paper. They may be adapted to the most exacting requirements.

You can get further information from your Du Pont salesman, or by writing to E. I. du Pont de Nemours & Co. (Inc.), Pigments Department, 1007 Market Street, Wilmington 98, Delaware.

DU PONT PIGMENTS for QUALITY PRODUCTION

Chrome Yellow ☆ Organic Yellow Lakes ☆ Soluble Blue
Pigment Green B—full strength and lakes ☆ Molybdate Orange
Dispersible "Monastral" Blue and Green Lakes ☆ Toluidine Red
Watchung Red ☆ PTMA—Blue, Green and Red Lakes

● Tune in to Du Pont "Cavalcade of America," Tuesday Nights—NBC coast to coast

DU PONT
PIGMENTS
FOR PAPERS

DU PONT
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BETTER THINGS FOR BETTER LIVING
. . . THROUGH CHEMISTRY

B&W Black Liquor Recovery Unit of 175 tons capacity at Calcasieu Paper Company.

An Example of B&W Engineering for Economy

CALCASIEU INCREASES PRODUCTION

...SERVED BY 4 NEW B&W UNITS

One of the most modern and up-to-date Kraft Mills in the country has recently been completed by the Calcasieu Paper Company, producing bag and wrap from log to finished shipment.

Chemical recovery and all steam-generating requirements at Calcasieu's modernized Elizabeth, La. plant are variously served by new B&W equipment. Part of a $6½ million expansion program, the following units are contributing to substantially increased daily plant capacity—from 60 tons in 1946 to 200 tons in May, 1950:

- *1* B&W Black Liquor Recovery Unit. Capacity: 175 tons, with 86,000 lb of steam per hr.
- *1* B&W Integral-Furnace Boiler, Type FJ, bark-fired, for generating up to 50,000 lb of steam per hr.
- *2* B&W Integral-Furnace Boilers, Type FJ. Capacity: 60,000 lb per hr each.

BABCOCK & WILCOX

B&W

B&W is proud that its equipment figures so prominently in this modern, highly integrated wood-use center . . . designed for its far-sighted management by the New York consultants, Alvin H. Johnson and Co., Inc.

P-756

The **BAGLEY & SEWALL**

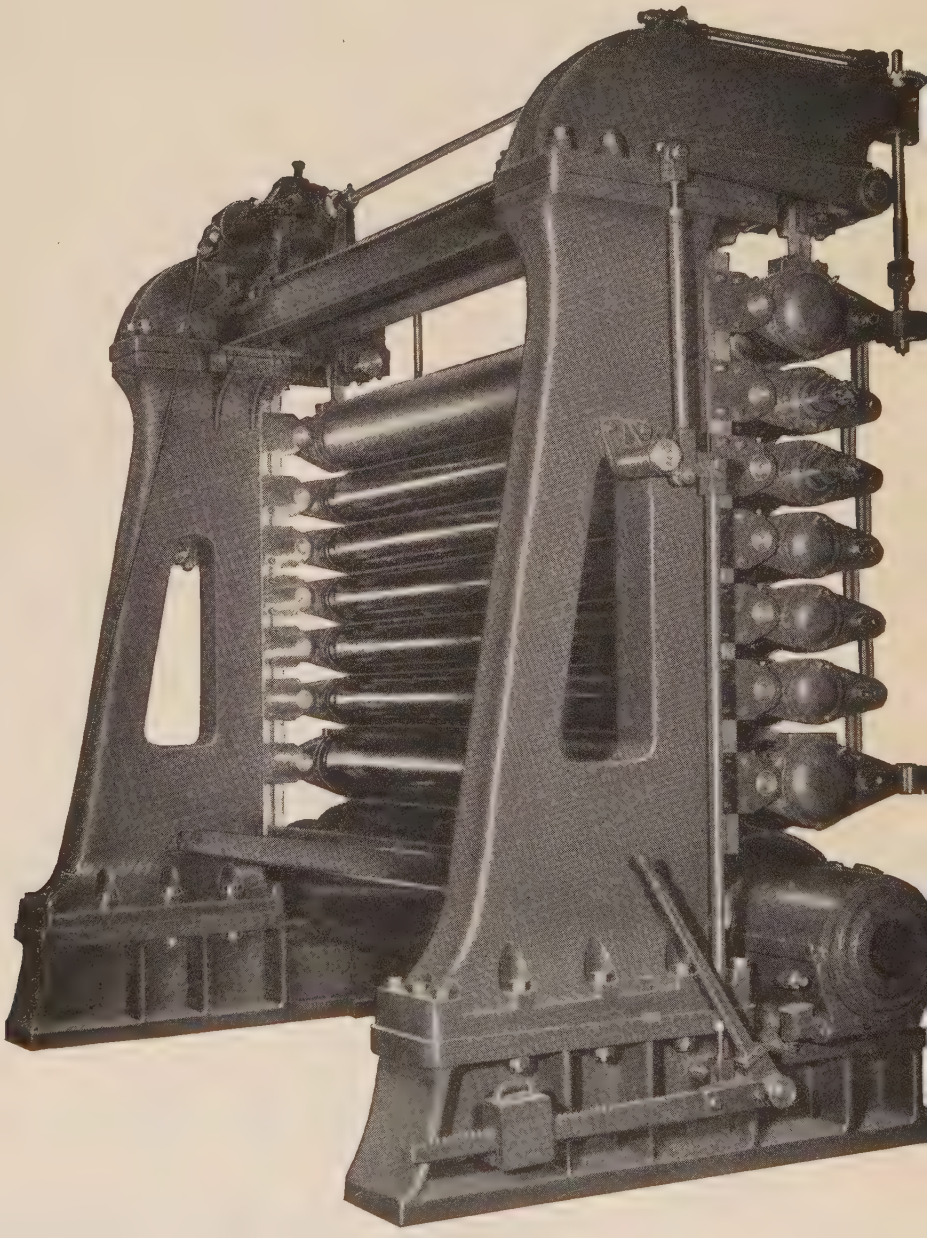
OPEN-SIDE CALENDER STACK

is is probably the easiest calender stack handle you ever used. All working parts in plain sight and easy to get at. You remove any roll in the stack by simply screwing four link shoulder bolts, reving two pivot pins, and out comes the . Simple as that!

a smooth-running stack. All the interdiate and top rolls are equipped with i-friction bearings, and each bearing using has a large-capacity oil reserve. e frame is solid — extra heavy construcn to eliminate vibration and to absorb avy impact loads.

e all Bagley and Sewall equipment, this ck was designed for long service and e of operation.

ite us when in need of a new calender ck. We're sure you'll like this one. It's initely "tops" in performance.



BAGLEY & SEWALL

DESIGNERS AND BUILDERS OF PAPER MAKING MACHINERY

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Specialty COATING

GREASE PROOFING – MOISTURE RESISTING – WATERPROOFING
MOLD RESISTING – REINFORCING – DECORATING, ETC.

is a job for WALDRON machines

Properties of Basic Packaging Papers

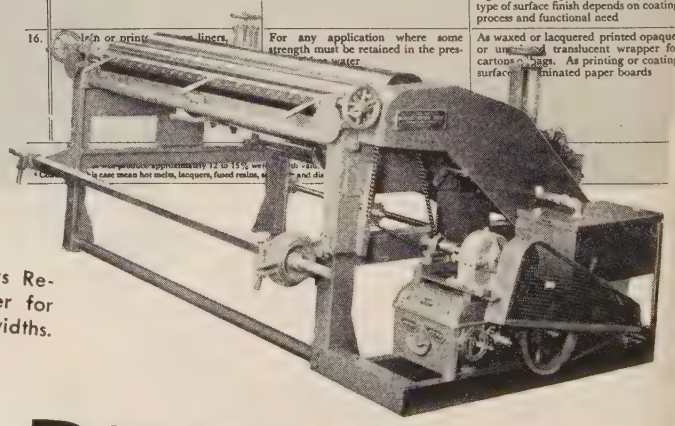
commercial standards. The six types ... are the ... ing. All of these papers can be coated and/or laminated to each other ... tional and decorative qualities desired. Manufacturers and converters are listed in the Buyers' Directory ... should be consulted for additional or for new uses of these materials. See also chart on *Properties of Metal Foils and Foil Laminations*, p. 205, and *Plastic Packaging Films*, p. 177.

ROPERTIES	KRAFT (SULPHATE)	GREASEPROOF	GLASSINE	VEGETABLE PARCHMENT	WET-STRENGTH[2]	BASE PAPERS[4] (For Coating or Waxing)
1						
ype of pulp or base ock used	Kraft (sulphate) pulp only	Hydrated sulphite ... sulphate	Glassine	1. High alpha cellulose from sulphite or	Usually sulphate ... with added	Sulphate or sulphite or mixed pulps
Weight range[1]	25 to 9					
Thickness range	Appr... lb. of					ly 0.001 in. per 10 lb. of
Finish	Wet mach... ende...					...es of calendering or paper ...s
Clarity and color	Opac... brow... blea...					...ached (white)
sical Test Data						
Bursting strength (Mullen)—lb. per sq. in.	Not... wet					...0% of basic weight
Tearing strength (Elmendorf)	Ve...					
Folding endurance	10...					
9. Wet strength	Lo...					
hemical						
10. Resistance to weak acids	Po...					
11. Resistance to alkalies	Poor					
12. Resistance to grease and oils	None	Good	Very good	12. Good to excellent on ... insoluble		None
Converting Characteristics						
13. Printability	Good, especially on well-sized bleached stock	Very good	Excellent	13. Very good with proper inks	Generally same as base-type paper, but may affect certain inks	Depends upon surface finish and printing process
14. Bag making	Good. Inexpensive, fast drying adhesives can be used for high-speed production	Good. Suitable for high-speed production with proper adhesives	Good. Suitable for high-speed production with proper adhesives	14. Requires special adhesives; relatively slow production on account of low surface penetration	Generally same as base-type paper, but may require adhesive modification	Good if coatings are heat sealable, or with proper adhesives
15. Coatings and laminations	High finish, well-sized stock preferred on account of normal high penetration	Adaptable to all types of laminations and coating	Approximately same as greaseproof, but preferred where highgloss, superior printing and maximum greaseproofness are desired	15. Relatively difficult to laminate or coat because of low surface penetration	Generally same as base-type paper	Surfaces are adapted for efficient formation of surface films of waxes and coatings for moistureproof uses. Degree and type of surface finish depends on coating process and functional need
16. General uses	For single and multi-ply bags used as shipping means. For single and duplex bags of all sizes. As a base for many laminations to paper and plastic film. As a base for waterproof papers using wax or asphalt as saturants or laminants. As a liner or wrapper for cartons	As bags, carton liners or wrapper not usually coated or printed. As a lamination to other paper or board	As bags, carton wrappers or liners either plain, printed, coated or laminated. As a lamination to other papers or boards	16. ...h or print... liners,	For any application where some strength must be retained in the pres... water	As waxed or lacquered printed opaque or ... translucent wrapper for cartons ... bags. As printing or coating surface ... minated paper boards

[1] Based on ream of 500 sheets, 24 in. by 36 in. Area factor (yield) $\frac{432{,}000}{\text{lb. per ream}}$ = sq. in. per lb.
[2] Plain kraft, glassine, or greaseproof paper can be given wet-strength characteristics by adding suitable resins to the pulp.

Whatever the basic stock, the function or quality required, you can depend upon WALDRON engineers to provide the right equipment for meeting the most exacting standards. With longer experience, more extensive research, testing and manufacturing facilities, wider range of machine designs—WALDRON can help you to turn out any type of coated product—better and faster.

Chart reproduced thru courtesy Modern Packaging Encyclopedia 1950.



Waldron-Meadows Reverse Roller Coater for varying material widths.

Coating Machines
Creping Machines
Embossing Machines
Floor Cov. Machines
Gumming Machines
Laminating Machines
Printing Machines
Rubber Mill Machines
Wall Paper Machines
Waxing Machines
Winders and Slitters
Special Design Machines

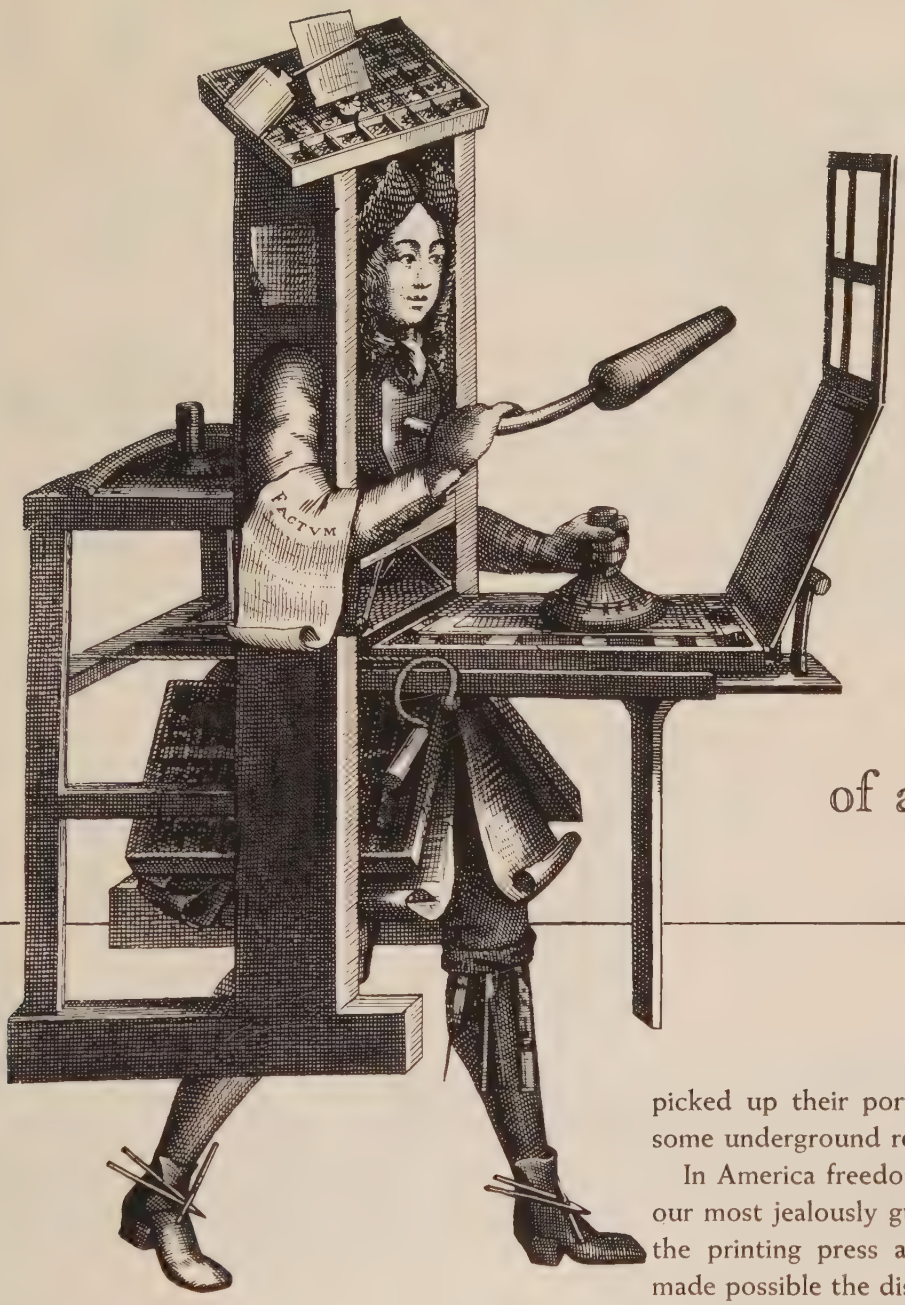
JOHN WALDRON CORP.

WALDRON DIVISION • MEADOWS DIVISION

Main Office & Works: NEW BRUNSWICK, NEW JERSEY

BUILDERS OF QUALITY MACHINES SINCE 1827

Illustration from the Bettmann Archive



Forerunner of a Free Press

Always the printing press has been an instrument for protest against tyranny, though often it has had to operate in secret. During the French Revolution, printers set up their mobile presses in streets and alleys and turned out pamphlets until, discovered by gendarmes, they picked up their portable plants and fled to some underground refuge.

In America freedom of the press is one of our most jealously guarded possessions. Here the printing press and plentiful paper have made possible the distribution of books, magazines and newspapers to all the people at little cost. Knowledge, culture and entertainment, spread without restriction, have helped to make and keep this a nation of free people with the highest standard of living in the world. Supplying this free press with its basic product—paper—is one of the great achievements of the American pulp and paper industry.

The dramatic story of paper is told in the sound-and-color film, "Paper — Pacemaker of Progress," and in a book under the same title. Both are presented by F. C. Huyck & Sons as a tribute to the Paper Industry. The book will be sent free upon request.

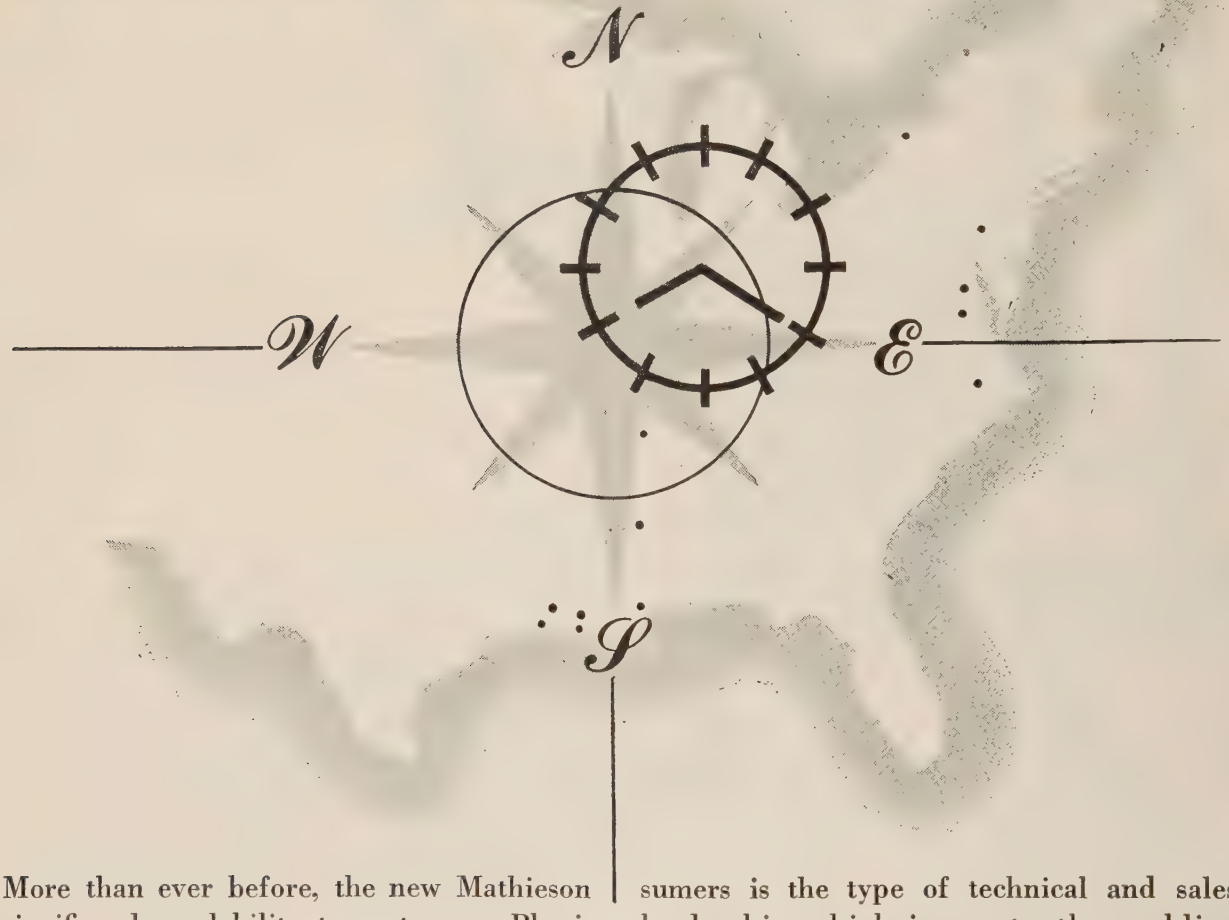
F. C. HUYCK & SONS · *Kenwood Mills* · RENSSELAER, N. Y.

KENWOOD WOOL PRODUCTS

HUYCK *Felts*

DEPENDABILITY

-that supplies heavy chemicals as needed



More than ever before, the new Mathieson signifies dependability to customers. Physically, Mathieson's 14 great plants, strategically located to serve America's chief industrial areas, turn out a wider array of heavy chemicals than any other producer in the country.

In addition, many of Mathieson's basic products can be combined in various ways to meet shifting market conditions. Equally important to chemical consumers is the type of technical and sales leadership which is constantly enabling Mathieson to raise its standards of service.

As a result, the new Mathieson affords dependability based on capacity to produce, flexibility of production, diversity of products and processes, and high standards of service. Mathieson Chemical Corporation, Mathieson Building, Baltimore 3, Maryland.

Mathieson CHEMICALS

12 Basic Heavy Chemicals • 14 Strategically Located Plants

SERVING INDUSTRY, AGRICULTURE AND PUBLIC HEALTH

Monel Winding Wire and Structural Rods help keep 'em running for a long time...

Your paper machines will last only as long as the metals in them.

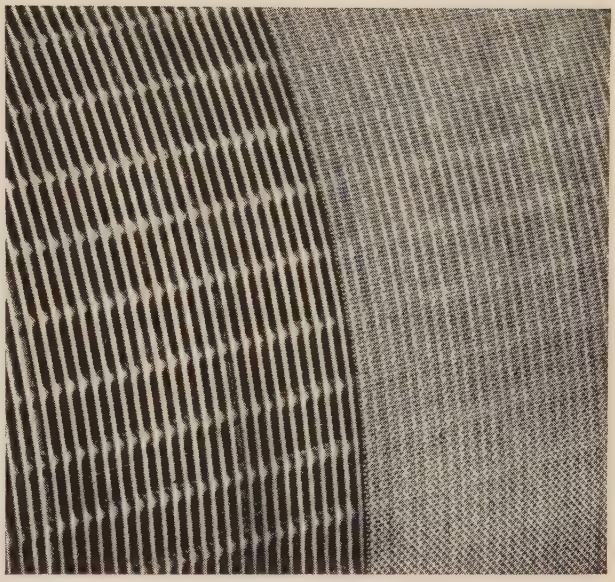
Which means, of course, that metals – today one of our most precious national assets – should be chosen with care.

On this page are examples of how one metal, Monel®, has been used to cut maintenance costs and extend the service life of paper and board-making equipment. Monel offers high strength and ductility, and exceptional resistance to corrosion by most commonly-encountered industrial chemicals. Monel is readily workable and weldable. It is produced in a variety of mill forms including wire, wire cloth, and fastenings.

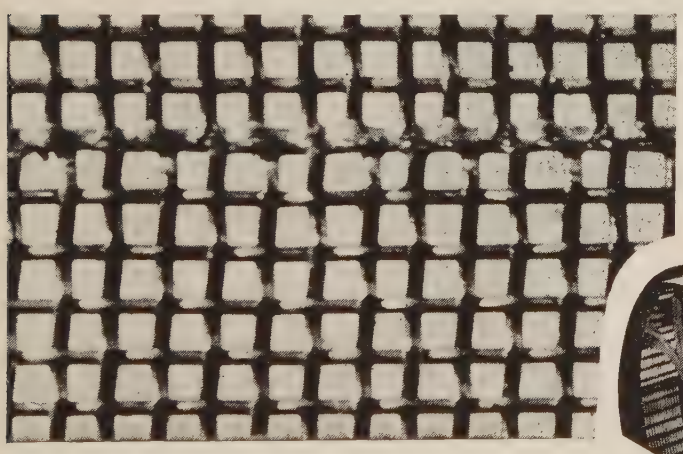
Because of current military needs, you may not be able to buy all the Monel you need, but INCO's Technical Service Department is always ready to help you solve corrosion problems.

Write for your copy of: *"How to Eliminate Unnecessary Shutdowns"*—written especially for paper mill men.

• • •



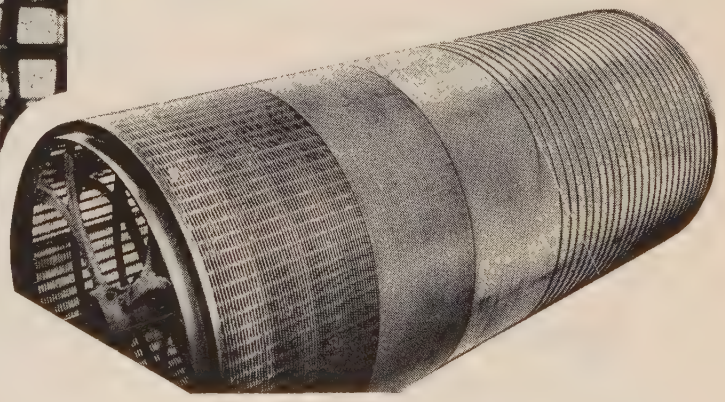
Monel Winding Wire. Cold drawn Monel wire is used over structural rods of cylinders, over drainage strips of drums, and as binding wire over wire cloth faces of filters. For this service, Monel wire is furnished with tensile strength of 110,000 to 140,000 psi. The endurance limit of Monel or its resistance to fatigue is much higher than most other non-ferrous alloys. It is so ductile that it can be wrapped around its own diameter without cracking. Where needed, smooth splices can be made by brazing or welding.



Monel roll covers resist both corrosion and mechanical fatigue, giving long and trouble-free service. Monel can be readily silver soldered. Monel cloth is produced in standard weaves, meshes, and widths.

Here are some typical service records:

1. **Soda Brown Stock Washer.** Monel cover still good after 22 months operation.
2. **Soda Pulp Thickeners.** 72 months service from one Monel cover; second in good condition after 25 months use.
3. **Sulfate Pulp Thickeners.** Monel has given 5 years service without failure on combined thickeners and washers.
4. **Sulfite Mill.** A mill which got 180 days service from bronze covers now gets 370 days service from Monel on #1 and #2 sulfite deckers.



Monel structural rods, used in drums, make strong, rigid, corrosion-resisting assemblies that need little attention. Monel structural rods have 26,000,000 psi modulus of elasticity, and 85,000 psi tensile strength.

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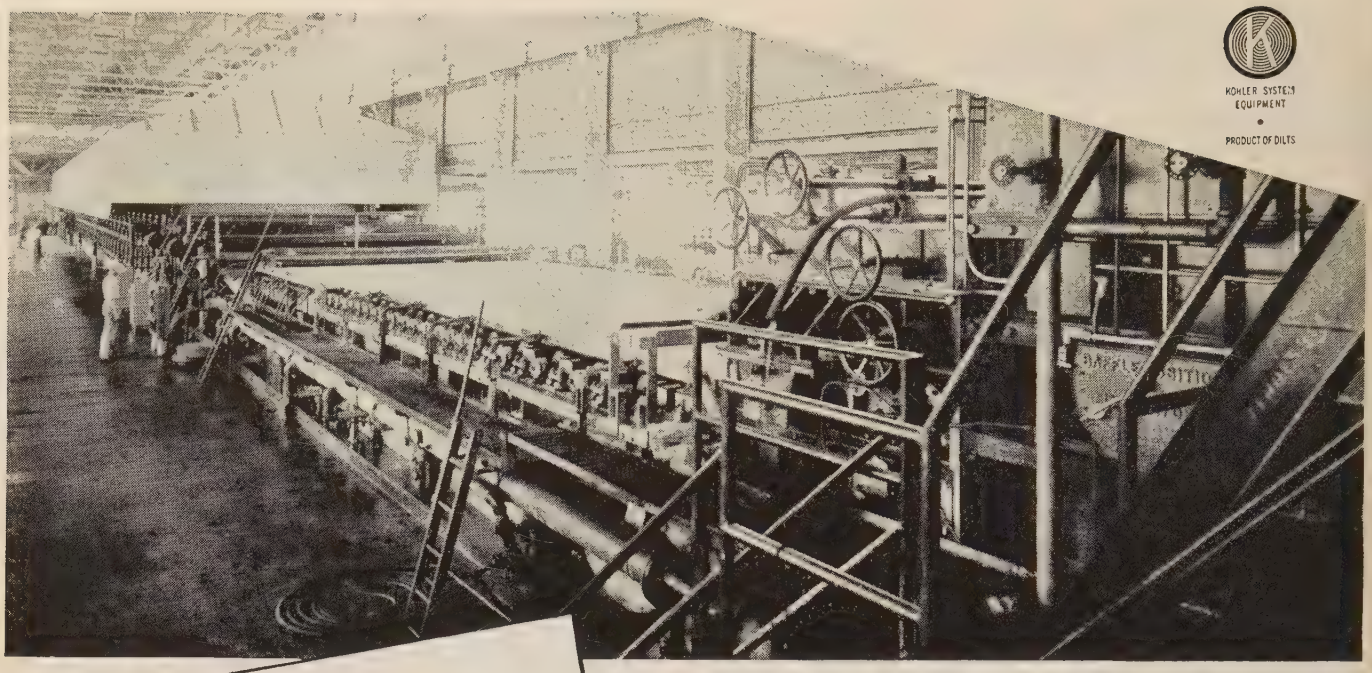
EMBLEM OF SERVICE
INCO
TRADE MARK

THE INTERNATIONAL NICKEL COMPANY, INC.
67 Wall Street, New York 5, N. Y.

MONEL... for Minimum Maintenance



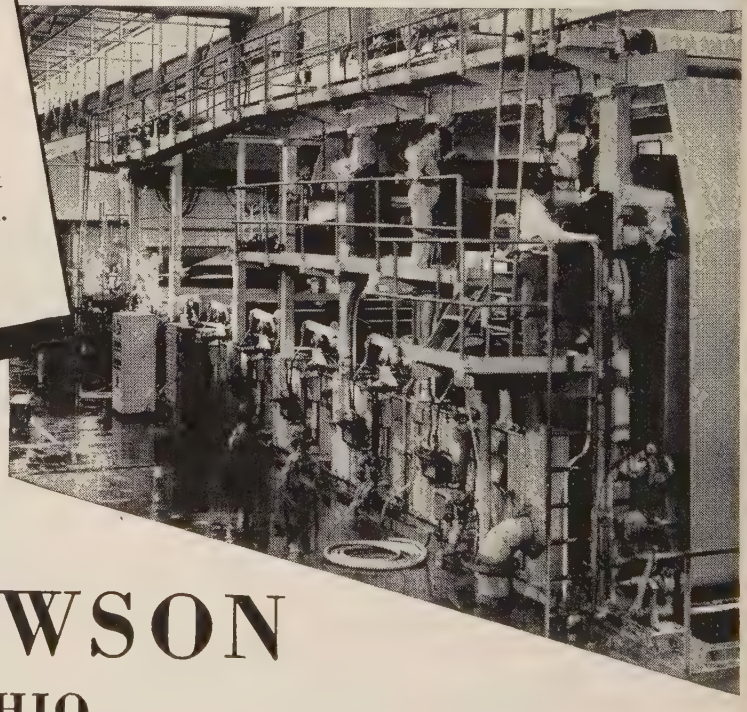
KOHLER SYSTEM EQUIPMENT
•
PRODUCT OF DILTS

Fourdriniers and Cylinder Machines

Fourdriniers that emphasize the extreme precision so necessary when running condenser and cigarette. Wide Fourdrinier for operation at high speeds.

Cylinder machines so carefully engineered, so painstakingly fabricated and so skillfully erected that they almost invariably make saleable paper within the hour.

Get the Black-Clawson estimate on that new machine or on your rebuilding program.



BLACK-CLAWSON

HAMILTON, OHIO

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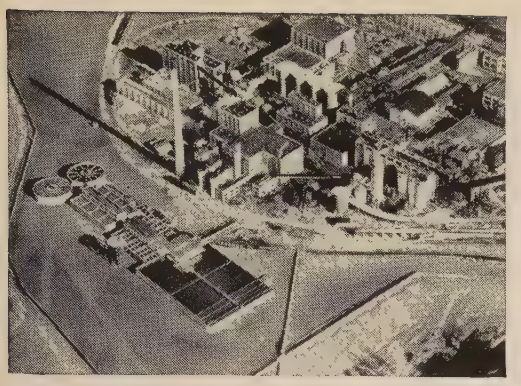

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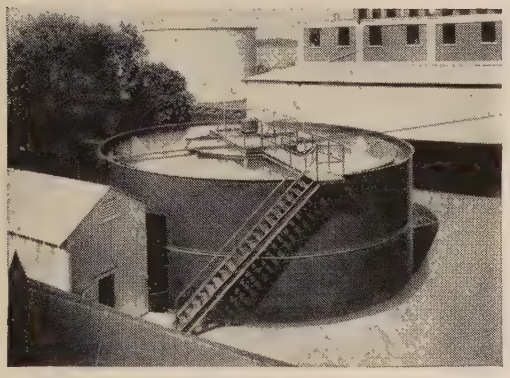

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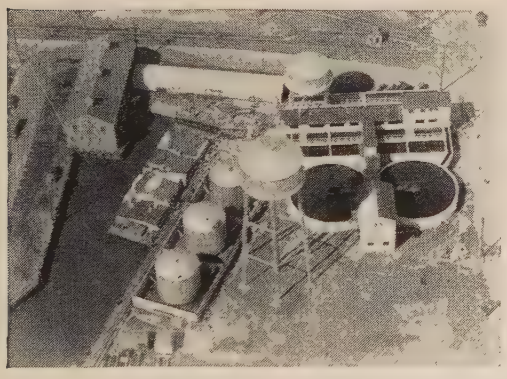

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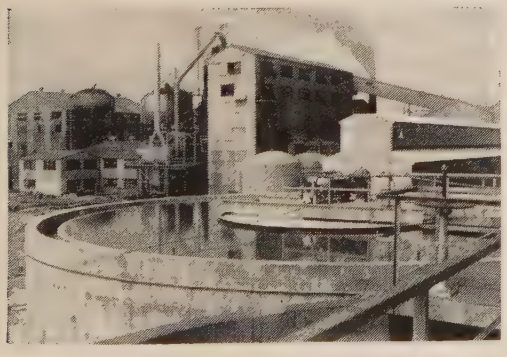

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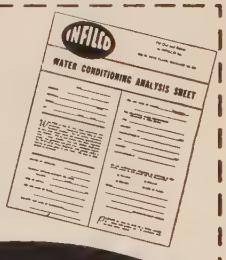

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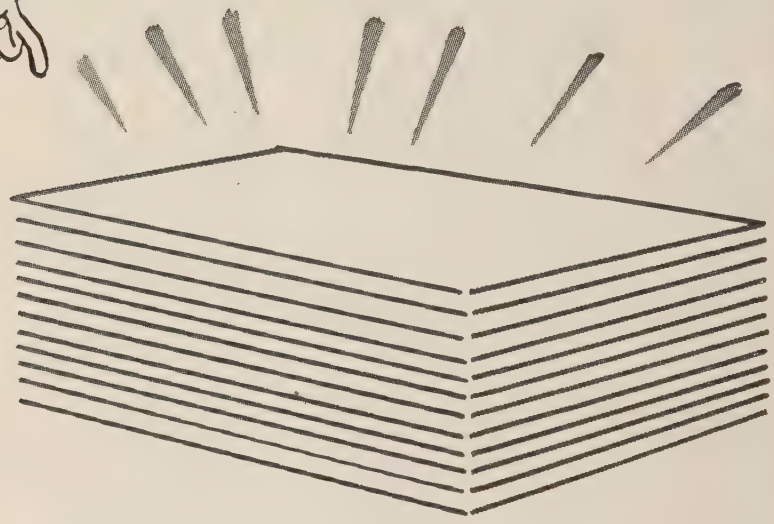

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The Effect of Compounding Variables on Styrene-Butadiene Latex Used for Protective Coatings for Paper

M. E. WENDT and WILLIAM H. AIKEN

A coating composition consisting of a butadiene-styrene copolymer latex, an emulsified wax, and a thickening agent was studied as a coating for paper. Emphasis was placed on obtaining a coated paper with good grease resistance and a low water-vapor transfer rate. The type of wax used has a considerable influence on the results obtained. Best results were obtained with high melting point microcrystalline waxes. At least 30 parts of wax solids per 100 parts of latex solids are necessary to give low water-vapor transfer rates. The use of a thickening agent improves resistance of the coating to passage of water vapor. Addition of thickening agents also reduces tendency toward foaming. The coating must be heated to a temperature of at least 180°F. to produce a continuous film of coating on the paper. Maximum properties were obtained by fusion of the coating at 300°F. On a 30-pound supercalendered kraft paper a water-vapor transfer rate of 1 gram per 100 square inches for 24 hours and excellent grease resistance were obtained in the laboratory with approximately 15 parts per ream of coating. The same results have been obtained with production equipment at half this coating weight. These properties were not appreciably affected by creasing the sheet.

Materials that are crystalline in structure and hydrophobic in nature are the most suitable members of the plastics family for imparting resistance to the passage of water vapor (*1*). Such materials as polyvinylidene chloride, rubber hydrochloride, and polyethylene meet these requirements, and all three are used extensively in the preparation of unsupported films for protective packaging and other uses. Unsupported plastic films are normally used in thicknesses of 1 mil and up in order to give sufficient strength and adequate protection.

Plastic materials may be applied to paper (*2*) by a variety of processes to give protective qualities to the sheet. Their use is somewhat limited by cost, because 15 to 40-pound per ream applications are generally required to give the desired properties. On the other hand, it has been found that excellent resistance to the passage of water vapor can be obtained with very thin coatings of some plastic materials by modification with small amounts of wax. On smooth, dense papers such as glassine, these coatings are effective at coating weights as low as 2.5 pounds per ream. Cellulose nitrate and cyclized natural rubber are the two plastic materials most widely used in conjunction with wax in this type of coating.

The mechanism by which the water-vapor transfer rate of a coating can be reduced by the addition of wax is not clear. It has been suggested that the wax migrates to the surface to form a thin film, and there is supporting evidence that this may be true, at least in the case of ethyl cellulose (*3*). The theory is also supported by the fact that the water-vapor transfer rate of most plastics—wax coatings—increases several fold upon creasing; however, part of this loss may be due to lack of flexibility in the plastic composition. The wax migration theory does not explain the increased resistance to the passage of water vapor brought about by the incorporation of wax in cyclized natural rubber, for both x-ray diagrams and the fact that the resin is soluble in hot wax indicates that the two materials are completely compatible (*4*). Whatever the mechanism, wax in combination with a plastic material is a much more effective water-vapor barrier than wax used alone.

The rapid expansion of the synthetic rubber and the plastics industries in the last 10 years has made available a number of materials of interest for treating paper. Many of these materials are produced by emulsion polymerization and are, therefore, available as aqueous dispersions. Such dispersions are usually referred to as latex because of their similarity in appearance and colloidal behavior to natural rubber latex. The latices of vinyl chloride copolymers, vinylidene chloride copolymers, polystyrene, butadiene-acrylonitrile copolymers, and butadiene-styrene copolymers are among the most interesting materials produced, and all are finding applications in industry.

Latices offer a number of advantages as paper coating materials. These include the absence of expensive, toxic, and/or inflammable solvents, and the ease of application on conventional paper coating equipment.

Of the various latices available, vinyl chloride copolymer (*5*) and vinylidene chloride copolymer (*6*) latices have been used most extensively to date in protective coatings for paper. Both of these materials offer good grease resistance at coating weights in the order of 20 pounds per ream, but to obtain water-vapor transfer rates of 1.5 grams per 100 square inches per 24 hours, coating weights in the order of 25 and 43 pounds per ream, respectively, are required.

The fact that incorporation of wax into resins will give good resistance to the passage of water vapor at light coating weights suggested that wax modification was worthy of investigation in latex systems. Of the various latices available, the butadiene-styrene copolymer latices appear to offer the most promise of success. These copolymers can be varied widely in butadiene-styrene ratio ranging from polybutadiene (a soft, weak rubber that is very compatible with wax) to polystyrene (a hard, brittle plastic that is incompatible with wax). Latices containing polymers with styrene contents up to 50% are manufactured in this country by the Rubber Reserve Corp. and are sold as GR-S. While

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the various GR-S latices may differ greatly from each other, they are all rubbery in nature, and all are compatible with waxes. However, the resultant coatings produced are too soft to be of general interest.

D'Ianni, Hess, and Mast (7) have investigated the physical characteristics of butadiene-styrene copolymers ranging in composition from 50 to 90% styrene. With increased styrene content, sheets prepared from the copolymers show increased hardness, tensile strength, stiffness, and heat distortion temperatures, but decreased elongation.

The latex of a polymer which contains 50% styrene will dry at room temperature to give a rather soft, rubbery film. As the styrene content is increased, the polymer loses its rubbery characteristics and harder plasticlike films are obtained. Still further increases in styrene content result in copolymers which are not film forming, but dry to give a resin powder rather than a continuous film.

Table I. Typical Properties of Pliolite Latex 170

Latex Properties	
Type latex	Butadiene-styrene co-polymer
Type stabilizer	Ammonium soap
pH	10.0
Mechanical stability	Good
Viscosity of latex (Brookfield), cp.	13
Solids, %	32
Surface tension, dynes/cm.	38
Dry Film Properties	
Specific gravity	1.01
Tensile strength, p.s.i.	900
Elongation, %	500
100% modulus, p.s.i.	170
Hardness—Shore A	76
Shore B	55

The latex chosen for this study was selected to give optimum properties of wax compatibility, film strength, and hardness. It is supplied commercially as Pliolite Latex 170 and typical properties are given in Table I.

PROCEDURES

Preparation of Coating Composition

In order to incorporate wax into the latex, it was necessary that the wax be in emulsion form. Waxes which are available commercially in emulsion form and waxes emulsified according to the procedure given below were both evaluated. It was observed that in order to give a viscosity suitable for coating and to prevent penetration of the coating into the paper, a thickening agent should be used.

Preparation of Wax Emulsion. Pliolite Latex 170 is stabilized with an ammonium soap, so that after drying of the deposited films, ammonia is lost to give a less water-sensitive film than would be possible if alkali-based soaps were used. Therefore, a volatile base soap—morpholine oleate—was chosen for emulsifying the wax. The ingredients used for preparing a wax emulsion of 50% solids were as follows:

Ingredients	*Parts (weight)*	*Per cent*
Wax	300	50
Oleic acid	17	2.84
Morpholine	13	2.16
Water	270	45.00

The wax was melted in a container that would hold 2.5 times the volume of the emulsion desired and brought to a temperature of 200°F. The oleic acid was added with stirring, and the temperature of the mixture brought back to 200°F. Morpholine was added slowly with stirring until the mixture became homogeneous and the temperature again brought to 200°F. The water was added at 200°F. with high-speed agitation which was continued until complete emulsification was accomplished. The emulsion was then stirred slowly until it had cooled well below the melting point of the wax. If air was occluded during mixing, the emulsion was allowed to age until free of air, then stirred slowly before use.

Preparation of Thickener Solution. Thickener solutions were prepared at 5% weight concentration. Enough warm water was added to thoroughly wet the thickener with care being exercised to avoid lumping. The remainder of the water was added as shaved ice or very cold water, and, after stirring the mixture, it was placed in a cold spot to age 10 to 15 hours, at which time solution was completed with vigorous stirring.

Preparation of Coating Composition. To prepare the coating composition, the wax emulsion was added slowly to the latex under mild agitation. When the mixture became homogeneous, the thickener solution was added, and agitation continued until the composition became smooth.

Preparation of Coated Paper

Laboratory samples were prepared by coating paper on one side with a Martinson coater which consists of a sliding bed plate which passes under a fixed knife. The coated sheet was then placed in a circulating air oven at 180° F. for 5 minutes to evaporate the water and fuse the coating.

While this method of coating appears to be satisfactory for comparative laboratory samples, it did not give results which correlated with those obtained in production equipment. In general, only half as much coating weight was required to get a given result in the commercial equipment when compared to paper coated in the laboratory.

Test Methods

Water-Vapor Transfer Rate. TAPPI Standard T 464 m-45 was followed with the exception that a relative humidity of 98% instead of 90% was used on the wet side of the samples.

Blocking Resistance. TAPPI Standard T 477 m-47 was used with the exception that a pressure of one third pound per square inch and a time of 4 hours was used.

Heat Seal. The coated paper was sealed face-to-face in an automatic heat sealer with a dwell time of 0.2 seconds, a temperature of 275°F., and a pressure sufficient to insure good contact. After cooling to room temperature, the sample was pulled by hand and judged for quality of heat seal, using the following ratings:

Excellent.....Tears the paper.
Good.....Tears fibers from the paper.
Poor.....Separates without tearing paper or fiber.

Oil and Grease Resistance. The sample was placed on a clean, white paper with the coated side up. A drop of oil or grease was placed on the coating, and the

length of time for it to spot through the coated paper onto the white paper was determined.

Crease Tests. Coated samples were creased prior to water vapor and grease resistance tests according to TAPPI Standard T 465 sm-44.

Foam Tests. The tendency for the coating composition to foam was measured by passing a controlled amount of air through a column of the coating and noting the maximum amount of foam produced and the time required to produce it. For this test, air was bubbled through 50 ml. of coating contained in a 500-ml. graduated buret with an inside diameter of 5.8 cm. Air was supplied from an air line. A tee connection in the air line bled out a portion of the air stream which was bubbled through water. Observation of the size and frequency of bubbles through the water provided a basis for adjustment of air input to hold the air flow at a constant rate. Test on a limited number of antifoaming agents on the latex by this method and in plant runs have indicated that fairly good correlation exists between results of this test method and behavior of the composition in plant equipment.

EFFECT OF TYPE OF WAX

The latex used in these studies gives films which in themselves are not highly resistant to the passage of water vapor. However, the polymer in the latex is quite compatible with petroleum waxes. In order to determine the effect of the type of wax on the properties of the coated paper, latex-wax emulsions were made by the procedure described above. Coating compositions were prepared, each containing a different wax in proportion of 50 parts of wax solids per 100 parts of latex solids. Except where otherwise specified, the coatings contained 2 parts of 1500 cp. methyl cellulose solids per 100 parts of latex solids. All of the waxes used were made into emulsions by the procedures described above with the exception of the Ceremul waxes which are supplied as emulsions. The properties of 30-pound supercalendered kraft coated with the various coatings are shown in Table II.

Superla wax gave a coating with the lowest water-vapor transfer rate and excellent grease resistance. Other microcrystalline waxes tested gave coatings having excellent grease resistance but water-vapor transfer rates more than double that of the coating containing Superla wax.

Since the Ceremul waxes were supplied in emulsion form, the type and amount of emulsifiers used in making the emulsions can very well account for the high water-vapor transfer rate and poor grease resistance obtained with these materials.

The low blocking temperatures may eliminate the use of the paraffin waxes for many applications. However, they may be of interest where cost is of great consideration and lowest values for water-vapor transfer rate and grease resistance are not required. It must be pointed out that even the 145°F. melting point paraffin wax gave a coated paper which had a slight block at 120°F.

Since little data were available concerning the purity and composition of the waxes used in this study, it was only possible to theorize as to type of wax which gives the most satisfactory latex-wax emulsion coating. Based on the limited knowledge of the waxes tested and the results obtained, it appears that microcrystalline waxes with a melting point of 169 to 190°F. produce the best coating with this latex.

Table II. Effect of Type of Wax

Type wax	Coating weight, lb./ream	WVTR, g./100 sq. in./24 hr. Unfolded	WVTR, g./100 sq. in./24 hr. Folded	Block temperature,[c] °F.[a]	Oil and grease resistance, hr. to spot: Olive oil	Mineral oil	Linseed oil	Cottonseed oil	Heat seal
Superla	14.0	0.90	2.11	NB-130	96+	96+	96+	96+	Excellent
Ceremul Q[b]	15.4	2.20	3.16	NB-130	96+	96+	96+	96+	Good
Nopco 1810R	14.0	1.92	3.01	NB-130	1	1	1	1	Good
AA-1144 wax	10.8	2.14	3.65	NB-130	96+	96+	96+	96+	Good
Amprol-23	15.0	2.06	3.03	...	96+	96+	96+	96+	...
Sun oil 1290	16.0	1.20	2.80	NB-130	96+	8	96+	96+	...
Magnowax-amber[c]	12.0	64.6	73.50	B-130	96+	96+	96+	96+	Good
Ceremul M[b]	13.0	10.50	14.20	SB-130	24	24	2	26	Poor
Be square	17.2	4.32	6.35	NB-130	..	..	..	..	Good
Ceremul C[b]	9.0	44.10	55.00	B-130	53	4	48	4	Poor
Ceremul R[b]	9.0	66.70	72.50	NB-130	29	32	1	4	Poor
Paraffin 134°F. m.p.	15.0	4.16	5.16	B-120°F.	13	13	96+	96+	Good
Paraffin 145°F. m.p.	15.0	2.05	3.75	SB-120°F.	96+	35	96+	96+	Good

[a] NB—no block; SB—slight block; B—blocked.
[b] Supplied in emulsion form.
[c] Thickened with Kelgin—All other coatings were thickened with 1500 cp. methyl cellulose.

EFFECT OF AMOUNT OF WAX

The effect of the amount of wax in the coating composition on the water-vapor transfer rate of coated paper was determined by varying the Superla wax content of a coating composition from 10 to 50 parts (dry weight) per 100 parts of latex solids. Two parts of 1500 cp. methyl cellulose on the weight of the latex was used as thickener. Table III and Fig. 1 gives results obtained on 30-pound supercalendered kraft coated with these compositions.

Table III. Effect of Amount of Wax

Type wax	Parts wax	Parts methyl cellulose	Coating weight, lb./ream	WVTR, g./100 sq. in./24 hr. Unfolded	WVTR, g./100 sq. in./24 hr. Folded
Superla	10	2	15	96.6	98.2
Superla	20	2	15	24.1	28.9
Superla	30	2	15	2.23	4.15
Superla	40	2	16.7	1.40	...
Superla	50	2	16.8	0.845	...

These tests showed that the water-vapor transfer rate of the coated paper is too high for the coating to be of practical value until at least 30 parts of wax are added. Increasing the wax content from 20 to 30 parts

brings the water-vapor transfer rate from very high values to a practical working range.

Table IV gives data on coating with 20, 30, and 40 parts of Superla wax per 100 parts of latex solids to which no thickener was added. The water-vapor transfer rates for coatings containing no thickener was slightly higher than those with coatings containing the equivalent amount of wax and 2 parts of thickener. Otherwise the data of Table III and Table IV show the same general trends.

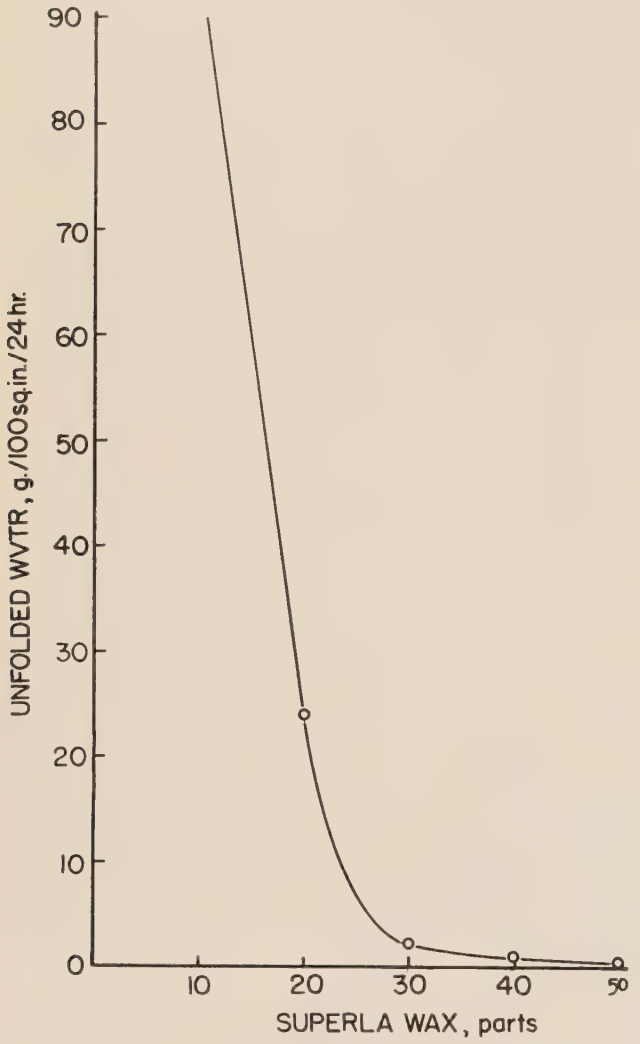

Fig. 1. Effect of amount of wax

Table IV. Effect of Amount of Wax (No Thickener)

Type wax	*Parts wax*	*Parts methyl cellulose*	*Coating weight, lb./ream*	*WVTR, g./100 sq. in./24 hr. Unfolded*	*Folded*
Superla	20	0	..	17.40	40.70
Superla	30	0	14.4	2.54	...
Superla	40	0	13.2	1.88	3.60

Based on these data as well as results obtained on paper coated on commercial equipment, it appears that a coating containing 50 parts of wax solids per 100 parts of latex solids should be used to give maximum grease resistance and the lowest water-vapor transfer rates. Greater amounts of wax tend to make the coating brittle and increase the water-vapor transfer rate of the paper.

It is interesting to note that contrary to results obtained on lacquer coatings containing wax, the water-vapor transfer rate of this coating is increased less than twofold by creasing.

EFFECT OF TYPE AND AMOUNT OF THICKENER

To study the effect of the type and amount of thickening agent on the viscosity of the latex-wax emulsion as well as the effect on the properties, coating compositions containing 100 parts of latex solids, 50 parts of Superla wax solids, and various amounts of 1500-cp. methyl cellulose and Kelgin were prepared. The coatings were applied to one side of a 30-pound supercalendered kraft paper and the resulting coated papers tested. The results are shown in Table V and Figs. 2 and 3.

The two thickeners behaved quite differently in their effect on the viscosity of the coating composition. Methyl cellulose did not greatly increase the viscosity of the coating until approximately 2 parts had been added, but upon the addition of further amounts the viscosity increased rapidly. Kelgin, on the other hand, gave large increases of viscosity with addition of small amounts, but as more Kelgin was added, the rate of viscosity increase was reduced. The two thickeners gave approximately the same viscosity at 2.9 parts of thickener. At lower thickener concentrations, Kelgin gave the greatest viscosity, but above 2.9 parts of thickener, methyl cellulose gave the greatest viscosity. The use of these data is important in order to arrive at coating compositions that will handle satisfactorily on specific types of coating equipment as well as to adjust the viscosity to give the desired coating weight.

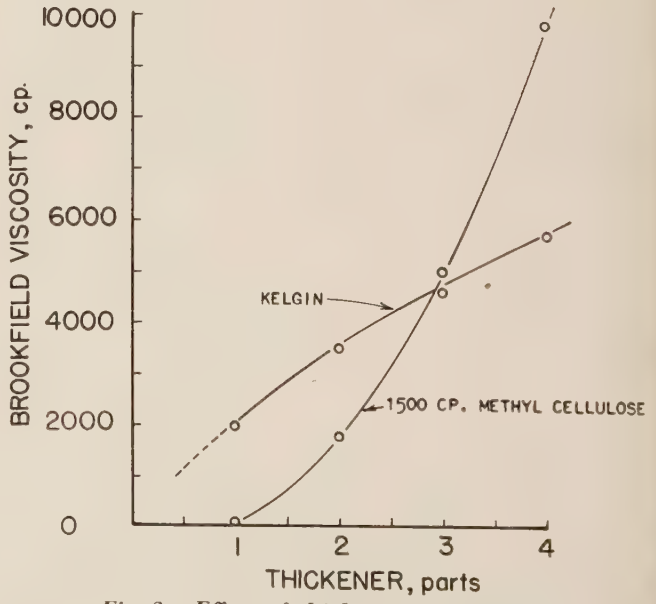

Fig. 2. Effect of thickeners on viscosity

Since methyl cellulose is more water sensitive than the latex solids or the wax, one might think that addition of this material would increase the water-vapor transfer rate. In these studies it was found that addition of methyl cellulose actually decreased the water-vapor transfer rate. It can be reasoned that by increasing the viscosity by the addition of thickener, a greater percentage of the coating applied to the paper remains

on the surface of the paper thus making a thicker continuous film. The addition of methyl cellulose appeared to have no effect on the blocking temperature or grease resistance of the coated paper.

Table V. Effect of Type and Amount of Thickeners

Thickener	Amount of thickener[a]	Brookfield viscosity, cp.	Coating weight, lb./ream	WVTR, g./100 sq. in./24 hr. Unfolded	WVTR, g./100 sq. in./24 hr. Folded	Oil and grease resistance, hr. to spot: Olive oil	Mineral oil	Linseed oil	Cottonseed oil	Block temperature, °F.
1500-cp. Methyl cellulose	1	107	13.0	1.42	2.41	96+	..	96+	96+	NB-130
1500-cp. Methyl cellulose	2	1800	14.0	0.90	2.11	96+	96+	96+	96+	NB-130
1500-cp. Methyl cellulose	2.5	..	16.4	0.86	1.47	96+	96+	96+	96+	NB-130
1500-cp. Methyl cellulose	3	5000	..	..	..	..	..	..	..	...
1500-cp. Methyl cellulose	4	9800	21.3	0.85	1.54	96+	96+	96+	96+	NB-130
Kelgin	1	2000	10.9	3.09	..	96+	96+	96+	96+	NB-130
Kelgin	3	4600	13.5	3.05	4.76	96+	96+	96+	96+	NB-130
Kelgin	4	5700	16.2	3.02	3.50	96+	96+	96+	96+	NB-130

[a] Parts per 100 parts of latex solids.

When Kelgin was substituted for methyl cellulose, the water-vapor transfer rate was more than doubled, and varying the amount of Kelgin did not greatly affect the water-vapor transfer rate of the coating. Although the coatings containing it were not equivalent in water-vapor transfer rate to those containing methyl cellulose, Kelgin did give coating compositions that had excellent grease resistance and a satisfactory blocking temperature.

Table VI. Effect of Coating Weight

Formula with 50 parts Superla: Coating weight, lb./ream	Formula with 50 parts Superla: WVTR, g./100 sq. in./24 hr., unfolded	Formula with 40 parts Superla: Coating weight, lb./ream	Formula with 40 parts Superla: WVTR, g./100 sq. in./24 hr., unfolded
Uncoated kraft	251.36	Uncoated kraft	251.36
5.0	3.73	..	....
8.6	2.19	7.7	4.25
13.5	1.16	13.4	2.32
16.8	0.85	16.7	1.40
21.9	0.64	21.8	0.95
26.4	0.70	26.4	0.97

EFFECT OF COATING WEIGHT

The effect of coating weight on properties imparted to the paper was determined on 30-pound supercalendered kraft paper coated on one side. The weight per ream of coating was varied from 7.7 to 26.4 pounds. Two compositions were testsd. Both contained 2 parts of 1500-cp. methyl cellulose per 100 parts of latex solids, but they differed in that one contained 50 parts of Superla wax and the other 40 parts of Superla wax.

The test results are shown in Table VI and in Fig. 4.

These data show that the water-vapor transfer rate was greatly decreased as coating weight increased until approximately 16 pounds per ream of coating had been applied. Above the coating weight of 16 pounds per ream the rate of improvement decreased. The data indicate that an increase in coating weight above 22 pounds per ream actually increased the water-vapor transfer rate. This increase at heavier coating weights appears to be caused by the formation of bubbles in the coating during the drying operations. These bubbles or blisters can be seen in the coating under a

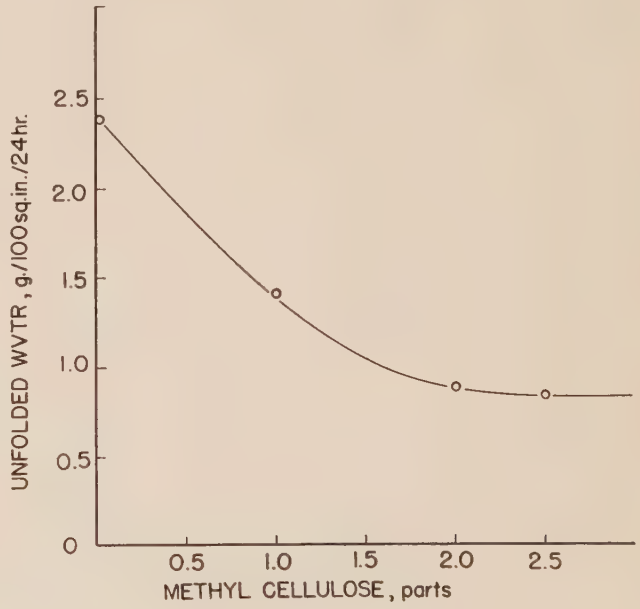

Fig. 3. Effect of amount of thickener

Table VII. Effect of Type of Paper

Type paper	Coating weight, lb./ream	WVTR, g./100 sq. in./24 hr. Unfolded	WVTR, g./100 sq. in./24 hr. Folded	Oil and grease resistance, hr. to spot: Olive oil	Mineral oil	Linseed oil	Lard
30-lb. Supercalendered kraft	5.0	3.73	4.88	..	..	..	..
30-lb. Supercalendered kraft	10.0	1.20	2.01	15	96+	96+	54
30-lb. Supercalendered kraft	16.0	0.862	1.47	96+	..	96+	..
25-lb. Glassine	2.9	0.856	1.62	96+	96+	96+	96+
25-lb. Glassine	5.6	0.422	0.890	96+	96+	96+	96+
25-lb. Glassine	9.0	0.350	0.680	96+	96+	96+	96+
25-lb. Supercalendered sulphite	1.8	47.6	54.4	..	..	..	16
25-lb. Supercalendered sulphite	4.2	3.53	6.25	..	..	..	..
25-lb. Supercalendered sulphite	6.1	3 67	4.89	..	..	..	40
50-lb. Glazed kraft	11.1	6.60	...	..	..	..	..
Chipboard	10.0	2.62	...	96+	96+	96+	96+

microscope. They are apparently caused by trapped water in the thick coating which is not released until the top surface of the coating film has been dried. These minute blisters reduce film thickness at the spot and, therefore, give a greater water-vapor transfer rate. The data in Table VI again emphasize the advantage of using 50 parts of wax over a lower amount.

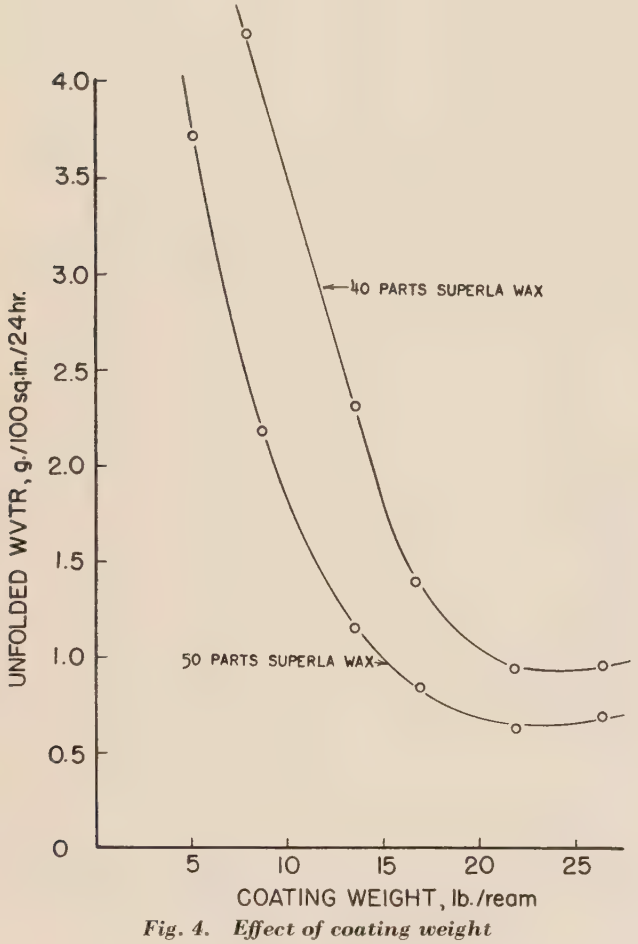

Fig. 4. Effect of coating weight

Although the data from these laboratory tests indicate that approximately 15 pounds per ream of coating must be applied to 30-pound supercalendered kraft paper, in order to obtain water-vapor transfer rates below 1 gram per 100 square inches per 24 hours, production runs on standard coating equipment have shown that water-vapor transfer rates in this range can be obtained with coating weights as light as 8 pounds per ream. It appears that the production coating equipment—along with immediate drying—leaves a thicker, more continuous film on the surface of the paper than is obtained with the coating and drying procedure used in the laboratory.

EFFECT OF TYPE OF PAPER

In order to eliminate the variable of paper type and finish, the experiments reported thus far were made using 30-pound supercalendered kraft paper. Table VII and Fig. 5 show results obtained by applying the coating to several types of paper. The coating consisted of 2 parts of 1500-cp. methyl cellulose and 50 parts of Superla wax to 100 parts of latex solids. These results indicate that the smoothness of surface to which the coating is applied is a determining factor in the water-vapor transfer rate that will be obtained with a given coating weight. Best results were obtained on glassine, followed by supercalendered kraft and supercalendered sulphite with results in the same order. The glazed kraft paper gave results somewhat poorer than those obtained with supercalendered kraft. The water-vapor transfer rate of the coating of chipboard was about double that of the coating on supercalendered kraft.

EFFECT OF ANTIFOAM AGENTS

As the components of the coating compositions are stabilized with soaps, they tend to foam when they are agitated before or during the coating operation. The foam in and on top of the coating is troublesome to handle. Problems from foam give the paper a poorer appearance, and more important, they leave a thinner coated area when the bubble breaks to increase the water-vapor transfer rate of the coated paper.

A number of antifoaming agents were tried in the coating in an effort to reduce foaming tendency of the coating. To a test formula consisting of 2 parts of 1500-cp. methyl cellulose and 50 parts of Superla wax per 100 parts of latex, 500 p.p.m. (on the wet weight) of various antifoam agents were added. The foaming

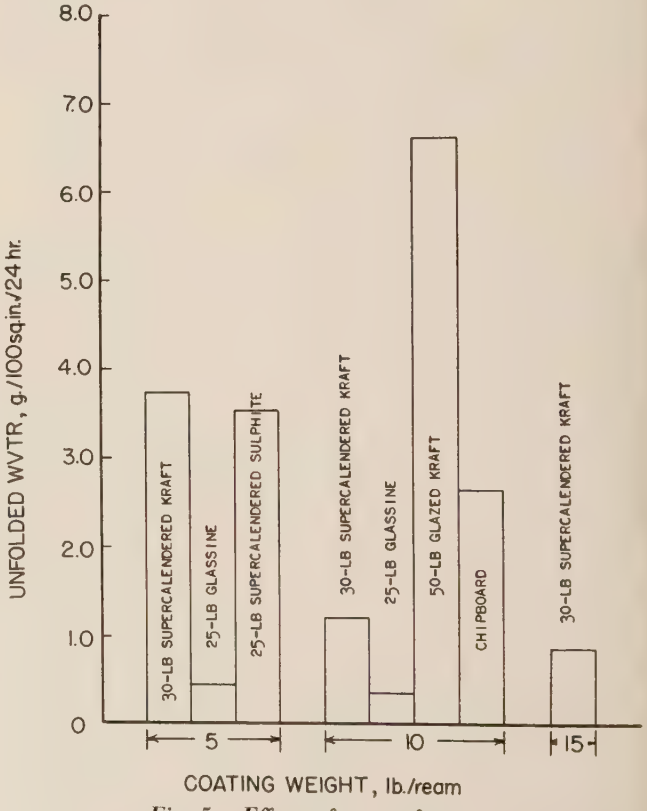

Fig. 5. Effect of type of paper

characteristics of these compositions as measured by the procedure described above are given in Table VIII.

Results on these compositions indicated that of the materials tested Dow Corning antifoam A is most effective in preventing foaming. The composition containing it foamed much less readily and less foam was produced.

The effect of amount of thickener upon foaming characteristics was determined by varying the amount of Kelgin and methyl cellulose in compositions containing

Table VIII. Effect of Antifoam Agents on Foaming

Antifoam	*Amount of antifoam p.p.m.*[a]	*Time required to raise to maximum level*	*Maximum level, cc.*
Dow Corning antifoam A	500	5 min.	75
Organosilicon 81066	500	45 sec.	100
Pine oil	500	2 min.	150
50/50 Foamex—DiGlycol Laurate S	500	1 min.	165
Foamex	500	2 min.	200
Poly glycol P1200	500	2 min.	225
Diafoam	500	2 min.	225
Octyl alcohol	500	1 min. 30 sec.	225
Aero—H	500	1 min.	425
De-Foamer—6	500	1 min.	500+
Nullafoam—S	500	50 sec.	500+

[a] Parts per million (wet weight).

50 parts of Superla wax solids per 100 parts of latex solids and 500 p.p.m. (on the wet weight) of Dow Corning antifoam A. The results are shown in Table IX.

With both thickeners, as the amount of thickener was increased the amount of foam produced was decreased and the foam was more difficult to form, thus it appears that by increasing the viscosity of the coating the foaming can be greatly reduced.

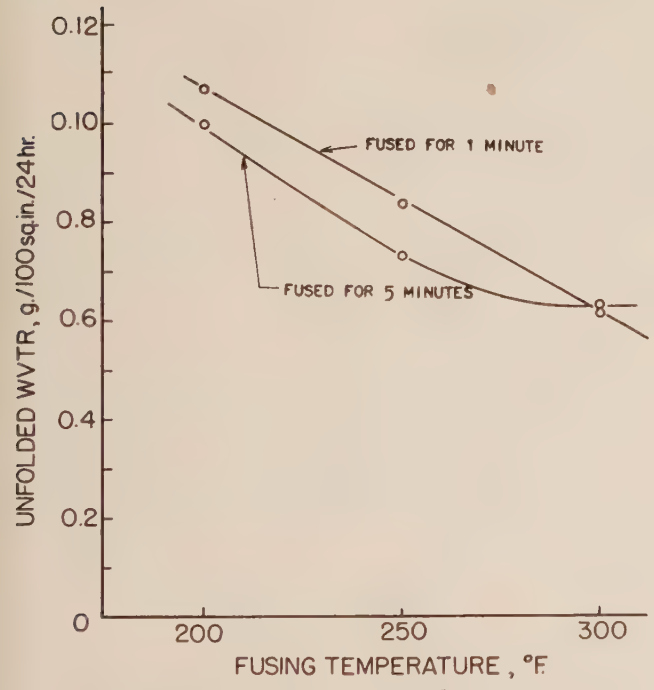

Fig. 6. Effect of fusing time and temperature

When the thickener is used in amounts less than 2 parts per 100 parts of latex solids, less foaming is obtained with Kelgin than with methyl cellulose for a given amount of thickener used. This is probably due to the greater thickening effect of the Kelgin. It appears that coatings thickened to a given viscosity with methyl cellulose would have less tendency toward foaming than those thickened to the same viscosity with Kelgin.

EFFECT OF FUSING TIME AND TEMPERATURE

If a continuous film is to be applied to the paper the individual particles of resin and wax in the latex-wax coatings must be fused at a temperature above the melting point of the wax. Without a continuous film, low

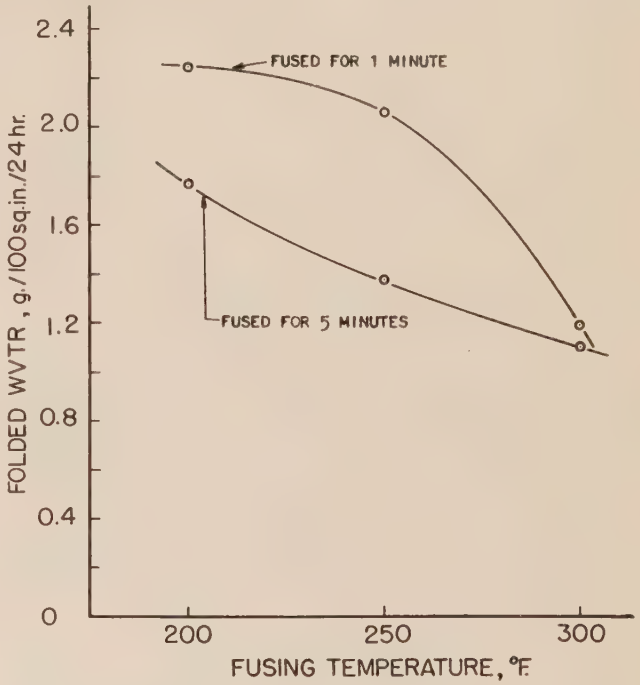

Fig. 7. Effect of fusing time and temperature

water-vapor transfer rates and grease resistance are impossible. To study the effect of fusing times and temperatures, supercalendered kraft paper was coated with 15 pounds per ream of a coating containing 2 parts of 1500-cp. methyl cellulose and 50 parts of Superla wax per 100 parts of latex solids. The coated paper was dried for approximately 5 minutes at 140°F. (which is below the melting point of the wax) immediately after the coating was applied. Different samples of the coated paper were placed in a circulating air oven for 1 and 5 minutes at 200, 250, and 300°F. Test results on these samples are given in Table X and Figs. 6 and 7.

Table IX. Effect of Type and Amount of Thickener on Foaming

Antifoam	*Amount of thickener, parts*	*Time required to raise to maximum level, sec.*	*Maximum level, cc.*
Methyl cellulose—1500 cp.	0.5	15	500+
Methyl cellulose—1500 cp.	1.0	25	500+
Methyl cellulose—1500 cp.	1.5	90	425
Methyl cellulose—1500 cp.	2.0	300	75
Kelgin	0.5	23	500+
Kelgin	1.0	120	350
Kelgin	1.5	120	100
Kelgin	2.0	120	90

At a fusion time of 1 minute an increase in temperature of 50°F. reduced the water-vapor transfer rate of the coating approximately 25%. At a fusion time of 5 minutes, increase in temperature also reduces the water-

vapor transfer rate. However, the water-vapor transfer rate for the samples fused for both 1 and 5 minutes at 300°F. were approximately the same. Thus it appears that with a fusion temperature of 300°F. the maximum properties of the coated paper can be obtained with a much shorter fusion time.

Table X. Effect of Fusing Time and Temperature

Fusing time, min.	Fusing temperature, °F.	Approximate coating weight, lb./ream	WVTR, g./100 sq. in./24 hr. Unfolded	WVTR, g./100 sq. in./24 hr. Folded	Block temperature °F.[a]	Oil and grease resistance, hr. to spot: Olive oil	Mineral oil	Linseed oil	Cottonseed oil
0	..	15	2.5	3.7	B-127	1 3/4	9	96+	1 1/2
1	200	15	1.07	2.22	B-140	96+	96+	96+	96+
1	250	15	0.835	2.07	SB-140	96+	96+	96+	96+
1	300	15	0.62	1.2	SB-150	96+	96+	96+	96+
5	200	15	1.0	1.78	B-150	96+	96+	96+	96+
5	250	15	0.735	1.39	SB-150	..	..	..	..
5	300	15	0.63	1.12	SB-150	96+	96+	96+	96+

[a] B—Blocked and SB—slight block.

The water-vapor transfer rates on the creased samples followed the same general trends as observed for the uncreased samples. Therefore, it appears that although the coating is very thin, it does not become brittle when exposed to the drying and fusion conditions employed in this study.

Comparison of fused and unfused samples shows that fusing is necessary to give good water-vapor transfer rates, good grease resistance, and high blocking temperature.

HANDLING PROCEDURES

Because the latex-wax coating is a collodial emulsion, it must be handled with some precaution to insure stability and to prevent excessive foaming.

It is suggested that the latex be placed in a glass lined or stainless steel tank (wooden tanks can be used although wood splinters must be removed from the coating) and a "Lightnin" mixer used to stir the latex. The mixer should be placed at an angle to prevent excessive vortex action on the surface of the latex. The wax emulsion should be fed from its storage container through a pipe which has its inlet below the surface of the latex. This will reduce the tendency to foam. After the wax emulsion is thoroughly mixed with the latex, the thickener solution and the antifoam agents are added and the stirring continued until the coating is a homogeneous mixture. Use of closed tanks and vessels are recommended to prevent loss of ammonia and, thus, the lowering of the pH of the latex and the coating. The latex and coating should be kept above a minimum pH of 9 and, morpholine or ammonia may be added if necessary to bring the pH up to this figure.

The coating can be fed to the coating station either by gravity or by pumping. Gravity feed is preferred as less foaming occurs; if pumping is desirable a triclover-type pump is recommended. It is suggested that valves that are easy to clean, such as the Saunder diaphragm valve, be used. To insure manufacture of a coating comparatively free from rust, wood splinters, etc., it is suggested that a screen be placed on the pipe inlet to the coating station. This pipe inlet should be placed below the surface of the coating of the pan so that foaming at this point is minimized.

If the coating pan is equipped with an overflow pipe, it is suggested that, if possible, the coating be returned to the storage tank by gravity. A triclover pump may be used, but in any case, the return line from the coating station should discharge below the coating level in the storage tank.

After a run has been completed the tanks and pipes should be flushed with softened water to prevent drying of the coating in the lines.

SUMMARY

A coating composition consisting of a butadiene-styrene co-polymer latex, an emulsified wax and a thickening agent was studied as a coating for paper. Emphasis was placed on obtaining a coated paper with good grease resistance and a low water-vapor transfer rate.

The type of wax used has a considerable influence on the results obtained. Best results were obtained with high melting point microcrystalline waxes. At least 30 parts of wax solids per 100 parts of latex solids are necessary to give low water-vapor transfer rates.

The use of a thickening agent improves resistance of the coating to passage of water vapor. Addition of thickening agents also reduces tendency toward foaming.

The coating must be heated to a temperature of at least 180°F. to produce a continuous film of coating on the paper. Maximum properties were obtained by fusion of the coating at 300°F.

On a 30-pound supercalendered kraft paper a water-vapor transfer rate of 1 gram per 100 square inches for 24 hours and excellent grease resistance were obtained in the laboratory with approximately 15 parts per ream of coating. The same results have been obtained with production equipment at half this coating weight. These properties were not appreciably affected by creasing the sheet.

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Chain Length and Chain-Length Distribution of Wood Cellulose as Compared with Cotton

EMIL HEUSER and LEIF JÖRGENSEN

Average chain length of and chain-length distribution in celluloses as they originally exist in lignified plant materials, such as wood, are still a matter of conjecture. Because no methods of isolating the cellulose component from such plant material without degradation are as yet available, we have to resort to compromises for the time being. A review shows which methods are available and what has been accomplished thus far. Attempts to improve the situation concern (1) limited pulping with bisulphite cooking liquors, (2) direct nitration, and (3) treatment with sodium chlorite in acid solution. Whereas the first method produces average D.P.'s for sprucewood cellulose which would seem to approach those as they originally exist in this wood species, neither of the other two methods produces acceptable results for this type of wood. However, when applied to aspenwood, the two methods yield values which would seem not to deviate too far from the original state. The possible reasons for the different behavior of the two types of wood are discussed. Raw cotton, grown under protection against atmospheric influences and taken from an unopened boll shortly before flowering, was chosen as a representative of cotton cellulose as it originally exists in the plant. Its average D.P. was 50 to 60% higher than the values found for the wood species. It is probable that this difference is to a great extent the result of a natural cause. The results of attempts to find the chain-length distribution of the cellulose as it originally exists in lignified plant material are likewise influenced and obscured by the effects of the degradation encountered during isolation. Thus, a certain quantity of the very short chain material, as found in sprucewood cellulose, undoubtedly was not present originally but resulted from degradation during isolation. Likewise, the longest chains, found to be of considerable length in the isolated cellulose, were probably still longer in its original state. In the cotton cellulose, the short chain material is missing, which appears to be the main reason for its greater uniformity as compared with wood cellulose. However, the medium and long chains still display considerable variation in length—that is, a polymolecularity which thus must be assigned to the cellulose as nature produces it in the cotton plant.

IN SPITE of many efforts made in recent years to throw light upon the average chain length of cellulose as it originally exists in wood and other lignified plant material, this value is still more or less a matter of conjecture, for no method has as yet been devised by which cellulose can be isolated entirely without degradation. An undegraded cellulose is also the prerequisite for an attempt to find a satisfactory answer to the question of its original distribution of chain length since, under the influence of the degrading methods of isolation, a change in distribution must necessarily be expected. Making the probable assumption that cellulose in wood possesses a certain degree of polymolecularity, the methods of isolation, such as sulphite pulping, bring about an apparent homogenization, in that the longer chains become shorter and approach the average length of the medium long and the short chains (*1–3*). Under severe conditions of sulphite pulping, however, all chains are markedly attacked. Methods of isolation which involve oxidation may have a similar effect and if, simultaneously, the isolating agent exerts a dissolving effect upon the shorter chain molecules, as is the case with the alkaline pulping methods, the original chain-length distribution undergoes additional changes (*1*).

Such changes must also be expected from the use of a number of laboratory methods which, from time to time, have been suggested for the isolation of the cellulose constituent in lignified plant material. Nor have the hopes of isolating cellulose without degradation by biological methods as yet materialized. Although progress has been made in the use of lignin-destroying fungi insofar as a more efficient reduction of the lignin content has been attained, the cellulose emerges from this treatment in considerably degraded form (*4*).

Thus, as the matter stands today and until better methods of isolation are available, we have to resort to compromises.

THE DEGREE OF POLYMERIZATION OF WOOD CELLULOSE

Attempts to isolate the cellulose from wood with as little degradation as possible include laboratory versions of the commercial sulphite pulping process, as first suggested by Klason in 1903 (*5*) and as modified later by other investigators. There is also the method of Cross and Bevan (*6*) which isolates the cellulose by treatments with chlorine and the modification of this method by Ritter and Kurth (*7*), by which the total carbohydrate fraction (i.e., the "holocellulose") is obtained. The latter studies were preceded by those of Schmidt and co-workers in the early twenties (*8*), who used chlorine dioxide for the same purpose. Today, this reagent has been replaced by sodium chlorite in acid solution. The sodium chlorite technique, first used by Jayme (*9*), has been perfected in recent years as an analytical method by Wise and co-workers (*10*). Finally, there are the methods which use nitric acid as a means of isolating the cellulose from wood and other lignified plant material. This technique, which probably had its beginning in the studies of Baly and Chorley in 1895 (*11*), were revived by Jahn and Coppick (*12*) and further improved by Mitchell (*13*). The action of nitric acid of a given concentration converts the lignin of the wood into a mixture of nitrated

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lignin and lignin oxidation products. This mixture is soluble in methyl alcohol* and, hence, may be removed from the nitrated wood ("wood nitrate"), leaving the cellulose in the form of its nitric acid ester. The purified nitrate may be dissolved in ethyl acetate or acetone. The solution is used for viscosity determinations, from which the degree of polymerization (D.P.) may be derived. It is claimed that the nitration treatment, when phosphoric instead of sulphuric acid is used in the nitration acid mixture, leaves the cellulose practically undegraded, whereas it attacks the noncellulosic carbohydrates to such an extent that they become soluble (or at least partially soluble) in methyl alcohol and are thus entirely or partially removed, together with the nitrolignin and the lignin oxidation products, during the purification procedure. Thus, the final purified nitrate is believed to represent the cellulose constituent of the wood (i.e., it contains only a little lignin and none of the noncellulosic carbohydrates).†

On a cursory inspection, it would appear that, of the various possibilities for isolating cellulose from its original plant material with as little degradation as possible, a well-buffered solution of sulphur dioxide would produce the least degraded cellulose perparation, particularly if all the other conditions of pulping were so chosen as to give a very mild cook.

PULPING OF WOOD WITH BISULPHITE COOKING LIQUORS

One method which promised encouraging results is to interrupt the sulphite pulping process at an early stage—that is, when the cellulose is still protected by substantial amounts of lignin. In recent years, such a technique was employed by Wultsch (*14*) and by Gralén and Rånby (*3*).

When cuprammonium hydroide was chosen as a solvent for the viscosity (and molecular weight**) determinations, Wultsch found that the lignin content of the samples (from sprucewood) may be as high as 10%—i.e., about one third of the lignin content of the original wood—before the solubility of the samples became impeded. Samples of this kind were obtained from a commercial digester after about 9 hours of digestion with an acid containing 5.28% sulphur dioxide and 0.92% calcium oxide. During this time, the temperature was allowed gradually to rise to 142°C. The average D.P. of this sample was about 1900. It is not quite clear whether this value had been corrected for lignin content. Apart from this, it is probable that even this limited pulping action had caused some degradation of the cellulose. Thus, the average D.P. of the cellulose as it existed in the wood used by Wultsch was undoubtedly higher than 1900.

Gralén and Rånby reported practically the same D.P. for a sample of sprucewood pulp which had been taken from a commercial digester after 14 hours of digestion with an acid containing 5.95% sulphur dioxide and 0.86% calcium oxide and which had a lignin content of only 3.7%. During this time, the temperature was allowed to rise to about 130°C.

Substantial progress in the chain-length determination of cellulose in wood pulps was made possible through the application of the nitration method. Earlier studies were made by Rassow and Wadewitz (*16*) in 1923, by Schieber and others [cited by (*1*)] in 1939 and, in this country, by Atchison (*1*) in The Institute of Paper Chemistry in 1943.

In Gralén and Rånby's study (*3*), it was shown that samples of sulphite pulp taken from the digester when their lignin contents were as high as 23% yielded nitrates which were practically completely soluble in acetone. Such samples resulted from 9 hours of (total) pulping with an acid containing 6.0% sulphur dioxide and 0.85% calcium oxide. Of this time, 4 hours were allowed for raising the temperature to 100°C. This temperature was then maintained during the remaining 5 hours. It would have been interesting to compare the D.P. of this sample with those of the other similarly produced preprations. However, in this case Gralén and Rånby did not employ the viscosity method. Instead, they subjected the acetone solutions of their nitrates to ultracentrifugal sedimentation and diffusion measurements. These resulted in D.P.'s five to six times higher than those derived from viscosity measurements (either of the sample in cuprammonium or of the nitrate in acetone). However, the authors themselves are rather doubtful about these extremely high values, and similar doubts have been expressed by other investigators (*17*).

If we accept Gralén and Rånby's cuprammonium viscosity D.P. values as being reasonably correct, it is of interest to note that they are not higher than those which might be expected if the commercial pulping process were allowed to accomplish its final objective. Thus, for a commercial black spruce sulphite pulp of the Mitscherlich type (that is, a pulp resulting from a slow cook under mild conditions*), an average D.P. of 1900, as determined by the nitrate method, was reported by Jayme (*18*). Likewise, in the Cellulose Laboratory of The Institue of Paper Chemistry we found for a commercial product from a Wisconsin mill the value of 1980, determined by a modified nitrate method. Our sample had a lignin content of only 1.0%, and the pentosan content of 4.0% may be regarded as also being on the low side for this type of pulp.

On the basis of these results, it was thought that sulphite pulping as a means of isolating cellulose as nearly as possible in its natural state could possibly be further improved. In addition, it appeared of interest to compare the results with those which are obtained with the direct nitration of wood and with the action of sodium chlorite upon wood, using the same wood material for all experiments.

In all cases, a minimum of treatment was aimed at. This minimum was considered to have been achieved when the cellulose preparation yielded a completely or nearly completely acetone-soluble nitrate for the D.P. determination.

The wood used was black spruce, taken from the

* Methanol for the purification of "wood nitrates" was probably first used by Husemann and Schulz (*13a*).

† To what extent this claim actually holds has not yet been satisfactorily clarified. It is difficult to determine the contaminations in the purified nitrate, and attempts to denitrate the preparation leads to oxidation and further decomposition of the cellulose constituent (see also later discussions in this publication).

** Using the Staudinger function (*15*).

* Fourteen to sixteen hours total cooking time; a maximum temperature of 125 to 130°C.—in contrast to a so-called "quick cook" with 9 to 11 hours total cooking time and a maximum temperature between 142 and 146°C.

same lot from which the commercial Mitscherlich pulp had been manufactured.

A great number of laboratory cooks were carried out, making use of the experience of Maass and co-workers (*19*) and of Yorsten (*20*), according to which we confined ourselves to the lower ranges of temperture. In addition, we used a stronger and more highly buffered cooking acid and, as a base, the more readily soluble magnesia instead of lime.

D.P. Determinations

The pulps were converted into their nitrates essentially in the way as previously described (*1*). However, since then, the original technique has been improved in various respects, chiefly with the aim of producing completely or nearly completely acetone-soluble nitrates.*

Preparation of the Nitration Acid

The nitration acid was a mixture of 50% by weight of fuming nitric acid (94% HNO_3), 40% of 84–85% phosphoric acid, and 10% of phosphoric pentoxide. The acid was prepared by adding to the phosphoric acid, which had been cooled to about 5°C., the calculated amount of reagent-grade phosphorus pentoxide. The likewise cooled nitric acid was then added. No loss of nitrogen oxides (NO_2; N_2O_4) should-occur when these precautions are observed. The mixture was then rotated until the phosphorus pentoxide was completely dissolved. No difficulties were encountered in accomplishing this within a reasonable period of time. The resulting solution varied from a pale yellow to a water-white color. The acid was stored in the dark and in a cool place. It should be used within a few days.

Nitration Procedure

The cellulosic material must be in a fluffy condition, in which form it is easily penetrated by the nitration acid. If the original material contained hard lumps, it was disintegrated in water and the suspension converted into very thin handsheets. The sheets were then torn into small pieces. Portions were weighed and transferred to a glass-stoppered 125-ml. Erlenmeyer flask. The nitration acid was quickly poured into the flask (50 ml. for 0.5 gram of air-dry material), the flask stoppered and immediately vigorously shaken by hand for about 5 minutes. The bottle was then allowed to rotate (at room temperature, if this was 24°C. or in a water bath at 24 ± 1°C., if the room temperature varied unduly) from $1^1/_2$ to 2 hours or longer, the shorter time being sufficient for bleached pulps, cotton, cotton linters, and similar lignin-free materials. Experiments have shown that a longer time, even up to 5 hours, does not essentially change the average D.P. of the nitrate.

Acid Removal and Stabilization

The nitrated material suspended in the nitration acid was poured into a fritted Büchner funnel, and the acid was sucked off to a point at which the cellulose nitrate was barely covered with acid. To avoid air being sucked into the nitrate while there was still acid present in the material and thus to avoid degradation of the cellulose (*20a*), the Büchner funnel was removed from the suction flask, filled with ice water, immediately replaced on the flask, and sucked again to a limited extent. This was repeated several times; after the preparation had been washed with a great amount of ice water, it was transferred quantitatively to a 2% solution of sodium carbonate and stirred vigorously for not longer than 15 minutes. It was then collected on a fritted Jena crucible and washed thoroughly with distilled water. The crucible was then allowed to stand in distilled water for 3 days, with two or three subsequent washes per day. Bleached pulps and like materials do not need the methanol treatment mentioned earlier. In these cases, the nitrate may be dried to constant weight (about 5 hours at 40°C.) and stored in a vacuum desiccator.

In the case of unbleached pulps and, particularly, if the preparations contained larger quantities of lignin, the water in the nitrates was displaced with methanol and the preparations rotated in glass-stoppered bottles for 2 to 5 hours, depending upon the amount of lignin present. The nitrates of ordinary unbleached pulps usually lost their brownish color after one treatment. The nitrates of the preparations higher in lignin required two or more treatments and, with preparations which were very high in lignin content, the brownish color could not be removed even on repeated methanol treatments. In such cases, some further purification was required. This was achieved when the nitrates were dissolved in acetone for the viscosity determinations (see under "Direct Nitration of Wood").

Nitrogen Contents and Nitrate Yields

The nitrogen contents ranged between 13.2 and 13.4% as determined by the Dupont semimicro nitrometer (*21*). The nitrate yields for the lignin-free cellulosic materials were always higher than would correspond to this nitrogen range, namely, 181 to 182% instead of 175 to 177%. Similar discrepancies were observed by Davidson (*22*). According to this investigator, the amount of phosphorus retained by the nitrate (0.2 to 0.4%)—probably in the form of phosphoric acid ester—would not fully account for the increased yield (the atomic weights of phosphorus and nitrogen being 31 and 14, respectively). Rather, it would appear that the discrepancy could be accounted for if the nitrogen contents of the nitrates were from 0.10 to 0.15% too low (as a constant error). Davidson's yields for nitrates of 13.75% nitrogen vary between 182.2 and 183.6%. The matter needs further study.*

Viscosity Determinations

Weighed quantities of the nitrate were dissolved in known volumes of acetone. Portions were removed with a pipet and diluted with acetone to the desired concentration (0.1 to 0.2%). The viscosity measurements were carried out in an Ostwald overflow viscometer. A kinetic-energy correction, derived from the velocity gradient of the viscometer, as suggested by Schulz (*23*) was applied to the measured data. The specific viscosities were converted into intrinsic viscosities by using the Huggins equation (*24*, *25*) and, from the intrinsic viscosities, the molecular weights (D.P.'s,

* The improved technique was essentially the result of a comprehensive study carried out in the Cellulose Laboratory of the Institute by Wm. H. Shockley.

* This becomes particularly obvious when attempts are made to use the nitrate yield for calculating the cellulose content of the wood. Thus, Mitchell (*13*) has calculated from nitrate yields of 120 to 130% a cellulose content for western hemlock of about 70%, a figure which is undoubtedly much too high.

respectively) were derived by using the Staudinger function with such values for the Staudinger constant as had been determined by us with the aid of osmotic pressure measurements in a special study.† Thus, for the wood cellulose preparations dealt with in this study, a K_m value of 11.6×10^{-4} was used.

Bisulphite Cooks

The cooks were carried out in a 1-gallon stainless-steel autoclave surrounded by an oil or wax bath. The temperature was adjusted to ±5°C. The autoclave, filled with about 30 grams of wood in the form of air-dry wood meal* and about 3000-ml. cooking acid, was brought to the desired cooking temperature within 20 minutes by preheating it in a bath of higher temperature. (The 20 minutes, being the same time for all experiments, is not included in the time listed in column two of Table I.)

After the wood meal had been treated for the desired length of time, the whole autoclave was submerged in cold water to arrest the digestion procedure quickly. When the pressure had decreased sufficiently, the lid was removed and the autoclave was filled with cold water. The digested material was filtered, washed several times with water, left in water overnight, filtered, and washed again. Eventually, the water was replaced with acetone and, after filtering, the samples were allowed to air dry.

The pulp samples thus obtained were nitrated, the nitrates purified, dissolved in acetone, the viscosities determined, and the latter values converted into molecular weights and D.P.'s as described earlier. The acetone solubility was determined by centrifuging the acetone solution, collecting the undissolved residue, drying, and weighing.

Results

The results for a selected number of pertinent bisulphite cooks are listed in Table I, showing the temperature and time, the pulp yields (determined in the customary manner), and the lignin and pentosan contents of the pulps. The last two columns list the acetone solubility and the D.P.'s (as calculated for cellulose).

The two series of experiments listed in Table I are chiefly distinguished by the (total) sulphur dioxide concentration of the cooking acids—8.5% in series I and 11.8% in series II.

It is seen from Table I that the highest D.P.'s were 2375 in series I and 2380 in series II. It would seem that these results were obtained essentially because the maximum pulping temperature was kept below 100°C. (that is, at 90°C.). This required 24 hours for series I and 14 hours for series II, the shorter time being obviously the result of the stronger acid. It is probable that a somewhat longer time for series II would have produced a completely acetone-soluble nitrate. Otherwise, the pulps were very similar, their yields, as well as the lignin and pentosan contents, being nearly the same.

Table I. Pulping of Spruce Wood with Magnesium Bisulphite Cooking Liquor

Cook	*Temperature, °C.*	*Time, hr.*	*Pulp yield, %*	*Lignin, %*	*Pentosan, %*	*Acetone solubility of nitrate, %*	*D.P*
		Series I.	Acid: 8.5% total SO_2; 3.03% combined SO_2[a]				
1	72	96	80.3	15.2	7.6	97	2090
2	80	48	85.8	18.0	8.4	95	1910
3	90	24	83.7	18.5	8.8	100	2375
4	107	4.5	82.8	19.5	8.7	95	2120
5	107	7.5	74.8	15.9	8.0	100	2050
6	107	15	60.3	5.9	7.3	100	1745
		Series II.	Acid: 11.8% total SO_2; 3.03% combined SO_2[a]				
1	80	32	85.0	18.4	8.2	100	2300
2	85	12	91.0	19.5	9.7	93	2210
3	90	14	84.5	18.3	7.9	99	2380
4	105	1.5	83.3	18.1	7.4	99	2350
5	105	3.0	74.7	16.1	8.0	100	2220

[a] Standard procedures were used for preparing the cooking acids and for analyzing them for their contents of total and combined SO_2.

It is interesting to note the very pronounced influence of the pulping temperature upon the time: An increase of 15 to 17°C. reduced the time to a few hours, with little or no loss in D.P., as shown in series II. However, the higher temperature became more detrimental when the time was substantially lengthened.

If the favorable results obtained with such a short cooking time could be confirmed, it may be expected that even better results can be produced by using a still stronger acid at still shorter times. It should be added that the rest of the cooks carried out (21 out of a total of 32) revealed the same trend—namely, that acids high in total and combined SO_2, in combination with a low cooking temperature, produced the better results (i.e., greater acetone solubility and higher D.P.'s). Cooks which were made with sodium as a base were not comparable with those listed in Table I. However, sodium, either as neutral sodium sulphite or as sodium bisulphite, would seem to have a chance of producing results either equal or even superior to those obtained with magnesium bisulphite.

The highest D.P.'s thus produced by sulphite digestion are 2400 to 2500 units—that is, distinctly higher than those mentioned earlier. The retention of almost two thirds of the original lignin content would seem to indicate that the pulping process was rather mild and that the cellulose constituent was only slightly affected. The D.P. of nearly 2400 would thus possibly be not too far below that of the cellulose as it exists in the wood.

† Being prepared for publication elsewhere.

* Air-dried chips of black sprucewood were disintegrated in a Wiley mill and screened. The 20 to 40-mesh fraction was used in all experiments. The wood meal thus obtained was extracted for 24 hours with a 1:2 alcohol-benzene mixture and air dried. The composition of the extracted wood was as follows: Cross and Bevan cellulose, 45.8%; lignin (TAPPI method), 27.3%; pentosan (as xylan), 12.5%; mannan, 8.5% (the latter two constituents being determined by Institute methods).

However, the fact that the nitrate yields were always higher than they would be if all lignin and noncellulosic carbohydrates were removed indicates that a higher D.P. must be assigned to the cellulose as it exists in the wood, than the values shown by our best preparations. That the nitrates must be regarded as still being contaminated with lignin and noncellulosic carbohydrates has also been experienced by Bryde and Rånby (*26*).

The latter publication became known after completion of our study. Bryde and Rånby subjected sprucewood to a mild pulping action in a small glass autoclave, using an aqueous ammonium bisulphite cooking liquor. Some of their preparations had lignin contents of 18 to 19%—i.e., very similar to ours—and they likewise obtained completely acetone-soluble nitrates. The highest D.P.'s of their preparations (3200 to 3300), as derived from intrinsic viscosities and using the Staudinger function but a K_m value different from ours, were recalculated by us so as to be comparable with our values. They then ranged between 2300 and 2540—i.e., values which are quite similar to those which resulted from our pulping method. Bryde and Rånby consider their D.P. values to be those of the cellulose as it exists in the wood.

DIRECT NITRATION OF WOOD

The preparations which were obtained by direct nitration of various woods by earlier investigators (*2*, *12*) were either of comparatively limited solubility in organic solvents or of suspiciously low average D.P., or they showed both deficiencies. Better results were obtained by Mitchell (*13*). The best of Mitchell's preparations of isolated cellulose (obtained from southern pine and western hemlock woods) showed average D.P.'s of 2000 and 1800, respectively (as recalculated by us, using our K_m value). Mitchell obtained these values by treating samples of the various wood species in the form of shavings or wood meal at 28°C. with a nitration acid composed of 64% HNO_3, 26% H_3PO_4, and 10% P_2O_5, for 31 hours in the case of pine and for 24 hours for hemlock and by washing the thus obtained "wood nitrates" with cold and subsequently with boiling water and finally extracting the preparations with methyl alcohol. The two nitrates were 99% soluble in ethyl acetate and had nitrogen contents of 13.8%.

As stated by Mitchell, the principle of his nitration procedure is "to nitrate the cellulose (in the wood) instantaneously and completely prior to elimination of the lignin component; then by continued action of the nitric acid, the lignin is modified so that it can subsequently be dissolved away from the cellulose portion of the wood."

In the present study, we followed essentially Mitchell's technique, employing some modification of the purification of the nitrates and using acetone as the solvent for the viscosity (D.P.) determinations.

About 1 gram of air-dry wood meal (8 to 9% moisture content) was treated with 50 ml. of nitration acid at room temperature (20 to 25°C.) for different lengths of time.

The stabilization and purification procedures were carried out as described earlier under "D.P. Determinations." However—apart from the necessity of repeating the methanol treatment several times—further purification was required. This was achieved by dissolving the vacuum-dried nitrate (50°C.) in 50-ml. acetone and shaking it for 12 hours—i.e., until the limit of its solubility was reached. The undissolved portions were centrifuged off, dried, and weighed as described before. The clear solution was then slowly poured (with stirring) into 50 ml. of a 1:1 mixture of water and acetone. The precipitate, usually only slightly colored, was dried in a vacuum oven (5 hours at 50°C.) and redissolved in acetone. The solution thus obtained was used for the D.P. determinations.

Sprucewood and aspenwood* and two different nitration acids were used. The results are shown in Table II. In the first series, a nitration acid very similar to that used by Mitchell was employed. However, in no case was the acetone solubility above 90% and the D.P.'s were low. Obviously, the nitration time of 24 hours was too long since the D.P. increased when the time was shortened.

In series II, the acid used was that previously described for the nitration of pulps relatively low in lignin content. However, the results were no better. In both series, the nitrate yields, although being lower for the wood nitrates than for the bisulphite pulps, were yet too high, indicating that noncellulosic contaminants were still present. It would thus appear that black sprucewood behaves toward direct nitration in a manner different from the woods used by Mitchell (pine and hemlock).** The actual yields of our nitrates were 93 to 95.6%. The 5-hour D.P. value of 2500 could thus probably be regarded as approaching the average chain length of the cellulose as it originally existed in the aspenwood.

This view is in a way supported by the fact that the nitration acid of the compositions used in this work caused no degradation of an entirely lignin-free cellulose (i.e., of cotton linters) when such material was nitrated under the same conditions.

Table II. Direct Nitration of Wood

Time of nitration, hr.	*Acetone solubility, %*	*Nitrate yield, %*	*D.P.*
Series I. Sprucewood. Acid composition: 65% HNO_3, 25% H_3PO_4, 10% P_2O_5			
24	89	108	1250
15	90	109	1585
8	87	112	1910
Series II. Sprucewood. Acid composition: 50% HNO_3, 40% H_3PO_4, 10% P_2O_5			
16	93	110	1730
8	67	116	..
Series III. Aspenwood. Acid composition as for Series II			
24	98–99	96	780
17	98–99	96	1360
5	98–99	95.6	2500
2.5	95	93	2460

As is seen from the table, our results with aspenwood were much better than those obtained with sprucewood.

Three of the preparations showed an acetone solu-

* The aspenwood used was from a lot previously used by R. March (*34*). The following analytical data were taken from March's doctor thesis:

Cross and Bevan cellulose	50.3%
Lignin (TAPPI Standard)	18.4%
Pentosans (as xylan) (Institute method)	20.8%

** Mitchell has frequently stated that each type of wood needs its special acid.

bility of 98 to 99% and, whereas nitration time of 24 and 17 hours were obviously too long for preserving a high D.P., a period of 5 hours appeared quite suitable. On the other hand, a period of $2^1/_2$ hours was too short to give the desired acetone solubility. The Cross and

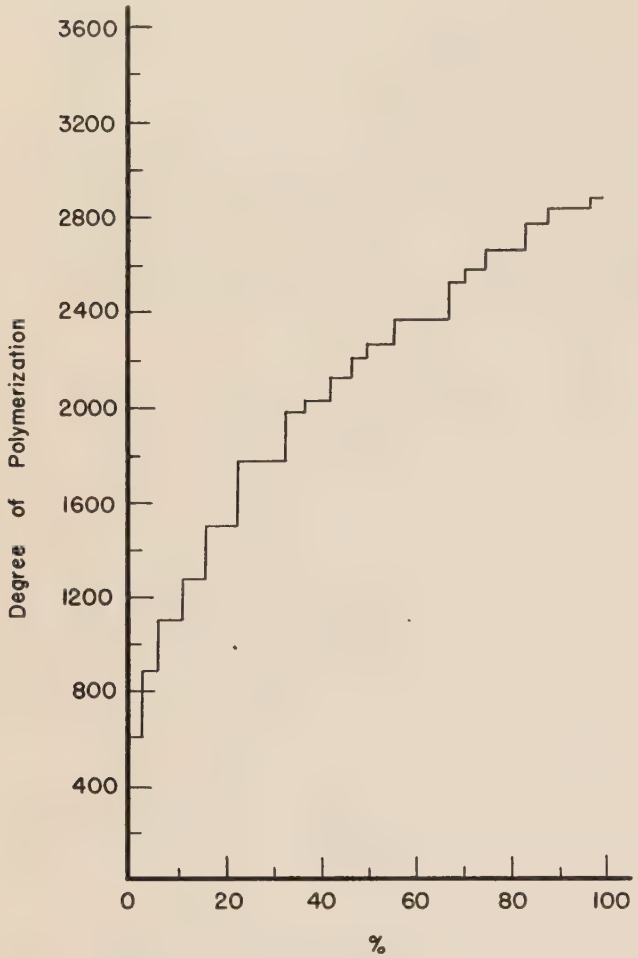

Fig. 1. Chain-length distribution of aspenwood cellulose (D.P. 2500)

Bevan cellulose content of the aspenwood was very close to 50%. This would call for a nitrate yield of 91.5%. This value is not too different from those listed in the table.

HOLOCELLULOSE FROM WOOD BY TREATMENT WITH SODIUM CHLORITE SOLUTION

Holocellulose was prepared from both the spruce- and aspenwood samples by the method of Wise and coworkers (*10*). The holocellulose preparations were converted into nitrates by the method described for the bisulphite pulps. The nitrates were dissolved in acetone and the D.P.'s calculated from viscosity measurements.

As indicated earlier, sodium chlorite is not a harmless delignifying agent. More recently it has become a general experience that it is difficult to remove the lignin from wood completely without degrading the cellulose, and that the degradation increases with increasing lignin removal. On the assumption that even substantial remainders of lignin and most of the noncellulosic carbohydrates would be removed from the holocellulose on nitration and subsequent purification of the nitrate, it was thought that to protect the cellulose against degradation, it would not be objectionable to leave in these preparations so much of the original lignin as would not too seriously interfere with the acetone-solubility of the corresponding nitrate. Whereas with the aspenwood we found this assumption in fairly good agreement with the experimental results, it did not apply in the case of the sprucewood.

Table III shows the results, as well as the number of sodium chlorites treatments given to the wood meal samples, each treatment comprising the use of 3.0 grams of chlorite per gram of air-dry wood in aqueous suspension at a pH of 4.0 to 5.0 and a temperature of 70 to 80°C. for 1 hour, 10 grams of air-dry wood meal being used for each experiment.

Table III. Treatment of Black Spruce and Aspenwoods with Sodium Chlorite

Number of treatments	*Holocellulose yield, %*	*Lignin, %*[a]	*Nitrate yield, %*	*Acetone solubility, %*	*D.P.*
Black spruce: original lignin content, 27.3%					
1	96.0	19.0	130	45	890
2	91.3	11.0	132	48	930
3	89.0	9.8	133	48	1090
4	83.4	4.5	139	90.5	1670
Aspen: original lignin content, 19.3%					
1	92.5	8.3	120	97	2410
2	87.0	0.5	130	97	1800
3	84.5	0.2	130	98	1785

[a] The values in this column are given with reservation since the lignin (according to a private communication from L. E. Wise) becomes so modified by the chlorite treatment that it no longer can be determined quantitatively by the Klason method. See also (*10a*) and (*10b*).

Again taking the acetone solubility of the nitrates as a criterion, we see that, in the case of sprucewood, even four treatments, which had reduced the original lignin content of 27 to 4.5%, gave a nitrate which was only 90% acetone soluble and the D.P. was only 1670. When more lignin was left in the holocellulose preparations, the acetone solubility dropped even below 50%. It looks as if, with sprucewood, even relatively small amounts of lignin in the holocellulose preparations interfere with the acetone solubility of the nitrate to a much greater extent than was observed with the bisulphite

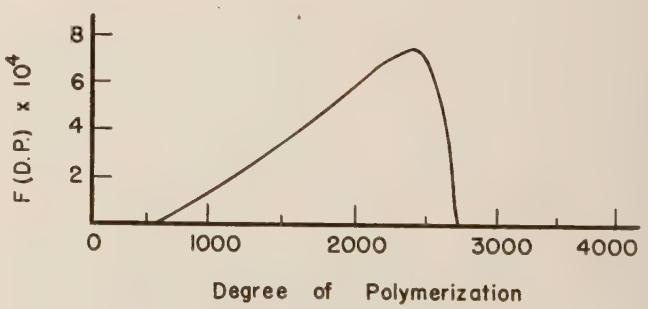

Fig. 2. Differential weight distribution of aspenwood cellulose

pulps and the wood nitrates as obtained from spruce wood. On the other hand, the nitrates from the aspenwood holocellulose preparations behaved again much better in this respect. It is seen that even an (apparent) lignin content of as high as 8% did not prevent the nitrate from reaching an acetone solubility of 97%.

Under these conditions, the average D.P. was about 2400. Further reduction of the lignin content in the holocellulose did not improve the acetone solubility of the nitrate but gave rise to considerable degradation of the cellulose constituent.

It would thus seem that, as far as aspenwood is concerned, some confidence may be placed in the D.P. value of 2400. This value is also of the same order of magnitude as that which resulted from the direct nitration of the aspenwood (2500).

Although both values are undoubtedly still below the D.P. of the native cellulose, they are probably not too far off, and we may conclude that, as far as aspenwood is concerned, both the direct nitration technique and the holocellulose method are capable of isolating the cellulose with only a small measure of degradation.

In connection with the isolation of the cellulose from sprucewood by means of sodium chlorite, it was thought of interest to determine whether the results obtained with the bisulphite pulps could be improved

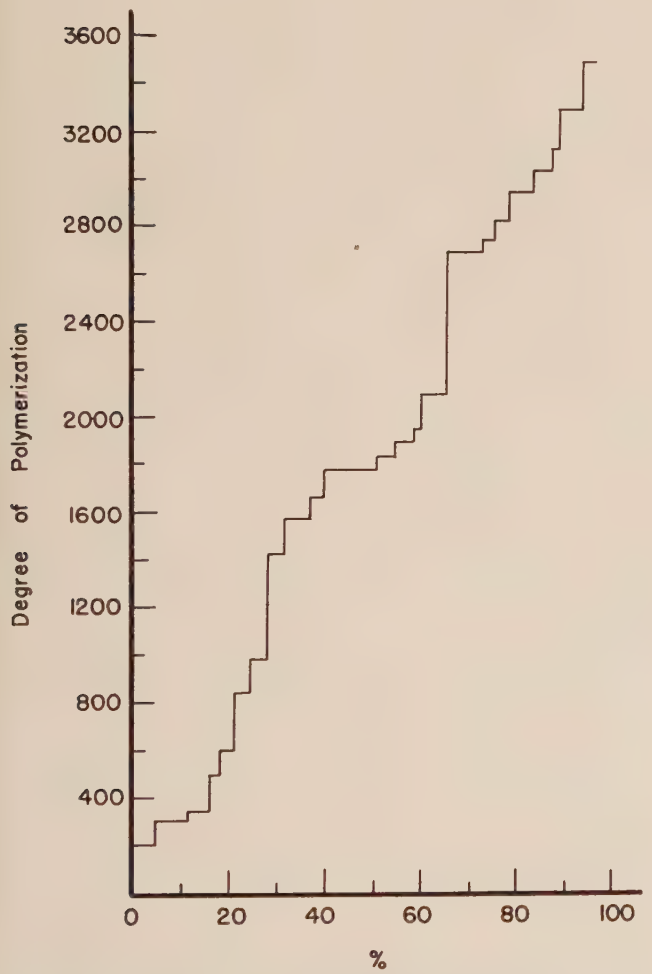

Fig. 3. Chain-length distribution of Mitscherlich pulp (D.P. 1980)

upon if, prior to nitration, their high lignin contents were reduced to low values by means of sodium chlorite treatment.

Accordingly, two of the bisulphite cooks (cooks 3 and 4 in Table I), as well as one other not listed earlier, all ranging in lignin content between 18.5 and 19.5%, were subjected to sodium chlorite treatments at 70 to 80° and 25°C. for varying lengths of time, details of which, together with the results, are listed in Table IV.

Table IV. Sodium Chlorite Treatment of Spruce Bisulphite Pulps

Sample	Time, hr.	Yield, %	Lignin (after $NaClO_2$ treatment), %[a]	Nitrate yield, %[b]	D.P.
Series I.	Bisulphite treatment: 24 hr. at 90°C., acid: total SO_2, 3.03% combined—magnesia base[c]				8.5%
	Original sample		18.5	119.2	2375
1	0.25	91.5	0.4	128.9	1940
2	0.5	87.8	0.1	132.0	1820
3	1.0	87.4	0.1	142.7	..
4	4.0[d]	97.6	3.2	121.4	2020
5	13.5[d]	95.1	0.8	133.2	2000
Series II.	Bisulphite treatment: 4.5 hr. at 107°C., acid as above[c]				
	Original sample		19.5	119.4	2120
1	0.25	93.6	0.8	126.5	1830
2	0.5	92.7	0.5	127.7	1790
3	1.0	83.6	0.0	137.4	1770
Series III.	Bisulphite treatment: 85 hr. at 80°C. acid: total SO_2, 1.95% combined—magnesia base				6.0%
	Original sample		19.0	119.0	2160
1	0.5	87.7	0.1	133.4	1920
2	1.0	84.6	0.1	139.1	1825

[a] See footnote to Table III.
[b] All nitrates were 99–100% soluble in acetone.
[c] See Table I.
[d] Temperature of $NaClO_2$ treatment, 25°C.

As is seen from Table IV, the lignin content was gradually reduced with increasing time of treatment but

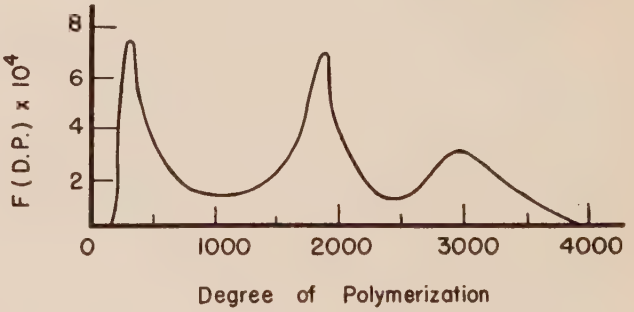

Fig. 4. Differential weight distribution of Mitscherlich pulp

the D.P.'s were likewise reduced. Although the latter reduction was less pronounced when the temperature of the sodium chlorite treatment was lowered to 25°C. with a correspondingly increased time (samples 4 and 5 of series I), in none of the experiments were the original D.P.'s of the bisulphite pulps (2375, 2120, and 2160, respectively) retained. Thus, in the case of sprucewood, nothing was gained by the sodium chlorite treatment of the bisulphite pulps, and bisulphite pulping alone would seem to be superior to both direct nitration and direct sodium chlorite treatment.

Whereas it is difficult at this time to give a satisfactory explanation for this different behavior of the two woods, the lower lignin content of the aspenwood and its different chemical composition (*27, 28, 28a*) certainly are factors to be considered. In addition, it is possible that, in aspenwood and, for that matter, in other hardwoods, with their shorter and less entangled fibers, the tissue is more easily penetrated by reagents and the lignin is thus more easily accessible than it is in spruce

and other coniferous woods. There are probably still other morphological differences which may help to explain the difference in behavior between these two types of wood.

THE DEGREE OF POLYMERIZATION OF COTTON CELLULOSE

The difficulties which are encountered in attempts to isolate the cellulose constituent without degradation from lignified plant material become negligible when the plant material is cotton. The few per cent of noncellulosic impurities (chiefly pectin and waxes) do not seem to affect the D.P., regardless of whether this characteristic is determined by the cuprammonium or the nitrate method.

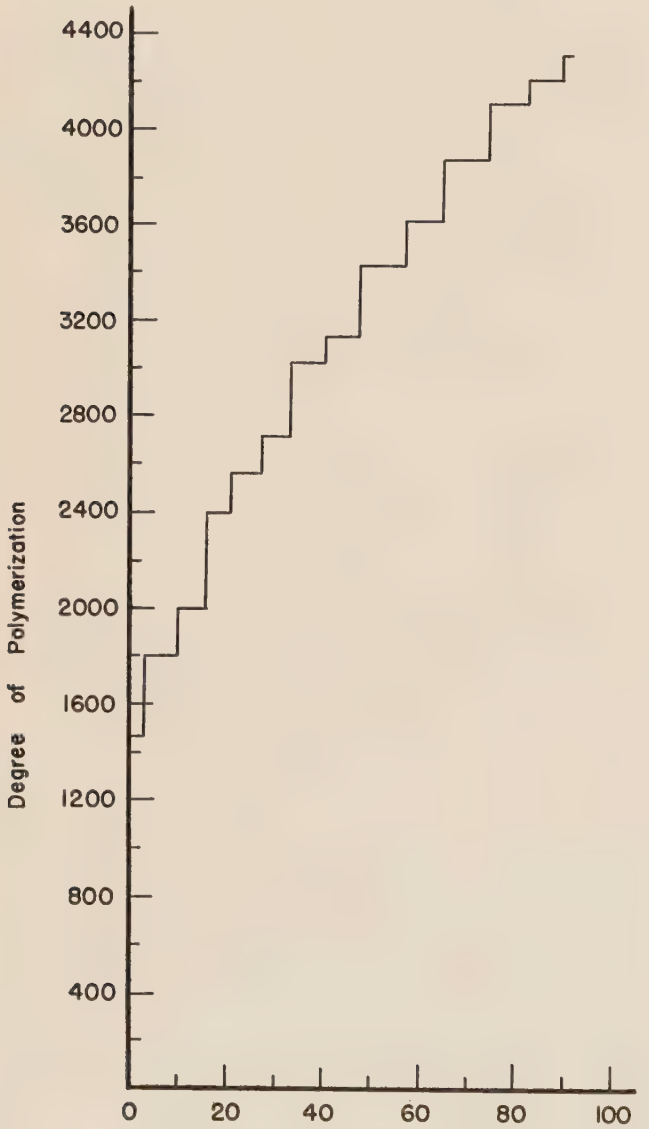

Fig. 5. Chain-length distribution of cotton from unopened boll

In most cases in which wood cellulose has been compared with cotton cellulose, cotton linters were selected as a representative of the latter. However, the indications are that nature builds up these shorter hairs also of shorter chain molecules—i.e., shorter than those which we find in the long-staple portion of the bolls. It is hardly necessary to mention that the cotton linters of the trade, since they usually have passed through purification and bleaching procedures, are more or less degraded, which makes them still less suitable for comparison, at least for the purpose of the present study.

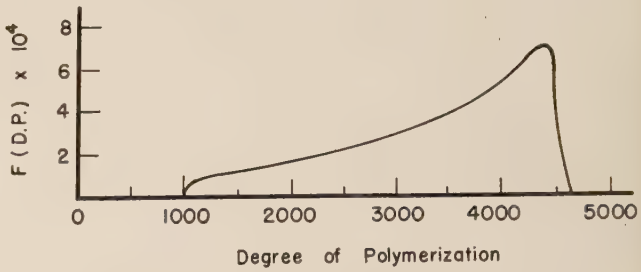

Fig. 6. Differential weight distribution of cotton from unopened boll

Our sample was an unbleached raw cotton, taken from the bolls just prior to maturing, which was at the 52nd day of flowering. The sample was marked "Stoneville 2B" and had been grown during the 1946 season on the grounds of the North Carolina Agricultural Experiment Station in Raleigh, protected from direct sunlight and other atmospheric influences. It was made available for our study through the kind cooperation of Thomas Kerr. After its removal from the bolls, the material was washed with hot water in order to eliminate enzymic effects during storage. The water was then displaced with cold acetone and the material allowed to dry in the air.

It is reasonable to believe that the precautions which were observed in obtaining this sample reduced degrading effects to a minimum and that the sample was thus representative of the long-staple portion of cotton in the state in which nature produces it.

The average D.P. of this material was determined by the nitrate method as described earlier. The K_m value used in the Staudinger function was 9.6×10^{-4} (based upon viscosity and osmotic pressure measurements*). An average D.P. of nearly 4000 was obtained.

This value is 500 to 1000 points higher than that found for cotton grown in the open and harvested at the customary time. On the other hand, our value is about 1000 points below the cuprammonium D.P. as reported by Jurisch (*29*) who likewise investigated a sample of cotton taken from the unopened boll. Although Jurisch does not name the source, it is probable that his was an Egyptian cotton since this type has repeatedly shown a higher average D.P. than the American type.

The values reported in the literature for unbleached cotton linters vary considerably. Thus, to give only two examples, Staudinger and Mohr (*30*) have listed a cuprammonium D.P. of 2700 and Mitchell (*13*) a (recalculated) nitrate D.P. of 2083 (2390 not recalculated).

Compared with the aspenwood cellulose sample reported earlier (D.P. 2500), the D.P. of the long-staple cotton is 1500 points or 60% higher. It is probable that differences of this order of magnitude will always be found when hardwood and cotton celluloses are com-

* This value remained unchanged during the period of observation—i.e., 7 months of storage in air in a dark flask and in a dark place in the laboratory

pared. As to sprucewood cellulose, the question remains still open and more experimental work has to be done.

The average D.P.'s of the various cellulosic materials discussed are summarized in Table V.

CHAIN-LENGTH DISTRIBUTION

Chain-length distribution data were ascertained for some of the cellulose preparations described in this study. Of the techniques most frequently used (*31–33*), fractional precipitation was selected because the results obtained therewith have been found to approach the true distribution more closely than those obtainable by fractional solution.*

The nitrates (purified as described earlier) were dissolved in acetone, water being used as the nonsolvent or precipitant. However, in order to divide the sample into as many fractions as possible and yet keep the volume of the solvent-nonsolvent mixtures within workable limits, the addition of water with subsequent centrifuging of the precipitate was practiced only for obtaining the first eight to ten fractions. From then on, precipitation was brought about by allowing portions of the acetone to evaporate from the mixture. This procedure produced an additional number of fractions. In order to obtain reproducible results, all conditions, particularly those of temperature and time for reaching equilibrium, were strictly controlled.

The precipitates were dried, weighed, and redissolved in acetone. The acetone solutions were used for the viscosity measurements, and the viscosities were converted into D.P.'s, as described in the first portion of this study.

Chain-length distributions were determined for:

(1) The aspenwood cellulose as isolated by the direct nitration method, having an average D.P. of 2500.
(2) The commercial Mitscherlich pulp, having an average D.P. of 1980.
(3) The cotton taken from an unopened boll, having an average D.P. of about 4000 (3930).

The aspenwood cellulose nitrate was divided into 18 fractions. The percentage weights and D.P.'s of these fractions are listed in Table VI.

Table VI. Fractionation of Aspenwood Cellulose (Av. D.P. 2500)

Fraction	*Weight, %*	*D.P.*
1	9.2	2860
2	4.6	2820
3	8.2	2760
4	4.2	2650
5	4.0	2570
6	11.4	2510
7	5.4	2360
8	3.6	2260
9	4.8	2200
10	4.8	2120
11	4.4	2020
12	10.0	1970
13	7.2	1775
14	4.8	1500
15	5.0	1270
16	3.4	1100
17	3.2	880
18	1.8	605

By plotting the percentage weights of the individual fractions (on the abscissa) against their average D.P.'s (on the ordinate), so-called step curves are obtained as shown in Fig. 1. These reveal the chain-length distribution at a glance. Table VI and Fig. 1 give an idea of the polymolecularity of the preparation. It is seen that the length of the chains range from as low as about 600 to nearly as high as 2900 anhydroglucose units. However, it is significant that there is no material of a D.P. below about 600 and that the material up to and including a D.P. of 1100 amounts to only about 8%. There is considerably more of a portion of medium length (namely, about 31%), ranging between 1270 and 2000. But the remaining material (about 60%) comprises rather long chains—i.e., with D.P.'s between 2100 and 2900.

Measured by the experience gained from chain-length distribution studies on other cellulosic materials, it would seem to be safe to say that the aspenwood cellulose has, indeed, suffered relatively little degradation during its isolation. If it had suffered to a greater extent, this would have shown up in the presence of con-

Table V. Average Degrees of Polymerization of Various Cellulosic Materials

Type of cellulose	*D.P.*	*Method of isolation*	*Investigator*
German sprucewood	1900[a]	Sulphite pulping, commercial conditions	Wultsch (*14*)
Swedish sprucewood	1900[a]	Sulphite pulping, commercial conditions	Gralén and Rånby (*3*)
German commercial Mitscherlich pulp	1900	Sulphite pulping, commercial conditions	Jayme (*9*)
Commercial Mitscherlich pulp	1980	Sulphite pulping, commercial conditions	This study
Black sprucewood	2380	Sulphite pulping with stronger and more highly buffered acid. Temperature below 100 °C.	This study
Swedish sprucewood	2300 to 2500	Pulping with ammonium bisulphite cooking liquor	Bryde and Rånby (*26*)
Southern pinewood	2000	Direct nitration	Mitchell (*13*)
Westerm hemlock	1800	Direct nitration	Mitchell (*13*)
Aspenwood	2500	Direct nitration	This study
Aspenwood	2400	Sodium chlorite, Wise, et al. (*10*)	This study
Cotton linters, unbleached	2700[a]	.....	Staudinger and Mohr (*29*)
Cotton linters, unbleached	2083	.....	Mitchell (*13*)
Cotton, long-staple lint from unopened bolls (probably Egyptian)	5000	.....	Jurisch (*28*)
Cotton, long-staple lint from unopened bolls	4000	.....	This study

[a] Cuprammonium method. All other D.P.'s were determined by the nitrate method.

Measured by the experience gained from chain-length distribution studies on other cellulosic materials, it would seem to be safe to say that the aspenwood cellulose has, indeed, suffered relatively little degradation during its isolation. If it had suffered to a greater extent, this would have shown up in the presence of con-

* Details will be found in a comparative study being prepared for publication.

siderable amounts of short chain material. Besides, there would probably be more of the medium long and less of the long and very long chain materials.

It is customary (*31, 1, 13*) to show the results also in the form of differential weight-distribution curves. These may be derived by interpolation from the integral weight-distribution curves—i.e., the smooth lines which may be drawn through the points of the step curves.

The differential weight-distribution curve of the aspenwood cellulose is shown in Fig. 2. It reveals the significant features referred to above—namely, the pronounced maximum in the high D.P. region and the absence of short chain material.

The fractionation data of the commercial Mitscherlich pulp are listed in Table VII. Twenty-three fractions were obtained.

Table VII. Fractionation of Commercial Mitscherlich Pulp (Av. D.P. 1980)

Fraction	*Weight, %*	*D.P.*
1	4.8	3500
2	1.3	3300
3	3.8	3130
4	5.4	3040
5	2.8	2950
6	2.6	2830
7	7.6	2750
8	6.0	2700
9	0.9	2100
10	4.5	1950
11	3.3	1900
12	11.1	1840
13	3.0	1785
14	5.6	1670
15	3.3	1580
16	3.8	1430
17	3.2	990
18	3.0	850
19	2.0	605
20	4.6	505
21	6.8	350
22	4.7	310
23	5.7	210

The picture here is quite different. The polymolecularity of the spruce cellulose, investigated in the form of a commercial Mitscherlich pulp sample, is much more pronounced. There is a considerable amount (nearly 24%) of short chain material below a D.P. of 600. Then follows a small amount (about 6%) of a portion comprising 850 to 990 units and a larger portion (34.6%) of material of medium length, ranging between about 1400 and 2000 units. The upper region (about 35%) extends to substantially higher D.P.'s than those found in the aspenwood cellulose, about one half ranging between 2950 and 3500 units.

This is also seen from the step curve (Fig. 3) and the differential weight-distribution curve (Fig. 4).

Considering that about 35% of the chains range between 2100 and 3500 and that about one half of this portion lies even between 3000 and 3500, the average D.P. of 1980 would appear to be low. But it is obvious that this low value results from the presence of considerable amounts of only medium long, as well as of short and very short chain materials.

The Mitscherlich pulp is a good example of the inadequacy of judging the behavior and performance of a cellulosic material solely by its average D.P. From this figure alone, it is impossible to surmise that this pulp actually contains such a relatively large portion of chains which are almost twice as long as the average D.P. indicates. Likewise, the average D.P. alone would not permit a prediction, with certainty, of the presence of such substantial amounts of very short chain material. Undoubtedly, it is the presence of considerable portions of both the very long and the very short chain materials which endows the Mitscherlich pulp with its special characteristics.

Because of the depressing effect which the short chain material exerts upon the over-all result, it is important to know whether this short chain material resulted from the pulping procedure or was originally present in the wood. If the former is the case, we would have to assign to the original sprucewood cellulose a very much higher average D.P. than 1980.

The chain-length distribution of the sprucewood cellulose which was obtained under milder conditions of sulphite pulping than that used in the commercial process and which had an average D.P. of about 2400 (see Table I), is still to be determined.

In the meantime, comparison may be made with the results which Bryde and Rånby (*26*) obtained with their best ammonium bisulphite cellulose preparations—i.e., of a (recalculated) average D.P. ranging between 2300 and 2500. Although these preparations had been obtained by a still milder sulphite pulping procedure than that used by us, they contained considerable percentages of short and very short chain materials. Therefore, one is inclined to believe that a considerable portion of the short and very short chain materials, as shown by both the ammonium bisulphite and the commercial Mitscherlich pulps, is inherent in the cellulose as it exists in the wood.

On the basis of our results, we likewise are inclined to believe that the characteristic differences which have been found for the celluloses from aspenwood and from sprucewood are real and that they may even hold for hardwoods and softwoods in general. Considering nature's preference for variety, this would not be surprising.

Table VIII. Fractionation of Cotton Cellulose (Av. D.P. 3930)

Fraction	*Weight, %*	*D.P.*
1	7.3	4300
2	8.0	4200
3	9.6	4100
4	8.0	3868
5	9.3	3620
6	7.1	3430
7	7.4	3130
8	6.2	3020
9	6.2	2720
10	5.1	2560
11	6.4	2400
12	6.7	2000
13	3.3	1800
14	8.4	1465

Table VIII shows the fractionation data and Fig. 5 the step curve for the cotton cellulose. It is interesting to note that not only the very short and the short chain materials, as shown by the Mitscherlich and the ammonium bisulphite pulps, are completely absent but also that certain portions of the medium long chains, which were found in the aspen wood cellulose, are missing in the cotton cellulose. The lowest range in the latter starts with a D.P. of as high as 1465. The per-

centage weights of materials of varying chain length may be grouped as in the other cases. Thus, as is seen from Table VIII, about 18.5% comprise chains between 1465 and 2000 units, about 24% between 2400 and 3020, and nearly 57% between 3100 and 4300. Of the latter, more than one half is in the uppermost range—i.e., between about 3900 and 4300.

The differential weight distribution of the cotton sample is shown in Fig. 6.

Although there is thus no question that even practically undegraded cellulose—that is, native cellulose in the form of cotton—is polymolecular, the polymolecularity of the wood pulps would seem to be more pronounced (particularly so for sprucewood cellulose).

The similarity between aspenwood cellulose and cotton cellulose is reflected in their differential weight-distribution curves (compare Fig. 2 and Fig. 6).

Although the present results do not furnish definite answers to our questions, we hope that they at least indicate how better answer may possibly be obtained.

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The Contribution of Sodium Silicate to the Stiffness of Paperboard

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The strength of unbonded plies of straw and kraft paper and of plies bonded with sodium silicate has been tested by several methods. The silicate adhesive considerably increases the rigidity as shown by ring crush, column crush, and stiffness tests. The bursting strength is decreased somewhat. It is more economical to increase the rigidity of paperboard by using additional sodium silicate rather than by using heavier paper.

It has been known for some time that sodium silicate adhesives increase the strength of corrugated boxes (*2*). If the contents are fragile it is especially important that the box be rigid and able by itself to support the weight of whatever may be piled on top. Perhaps the rigidity could be increased by substituting a layer of sodium silicate for some of the paper. This could be accomplished by applying the silicate adhesive in layers much thicker than are required for adhesion alone. Because of the relatively low cost of silicate adhesives this should prove economical.

A series of tests was made under controlled conditions to determine quantitatively the contribution of sodium silicate to the strength of laminated solid fiber. Combinations of two and three-ply straw or kraft paper with various spreads of silicate adhesive were compared.

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EXPERIMENTAL METHODS

Two types of paper were used: straw and sized

Table I. Sodium Silicate Adhesives

Trade name	*Ratio: silica/soda*	*Density*
Stixso DD	3.40	39.7° Bé
O	3.22	42.2

kraft. Both were 10 point liners, the straw weighing 38 pounds per 1000 square feet, and the kraft 44 pounds. Standard grades of sodium silicate were used; their properties are shown in Table I.

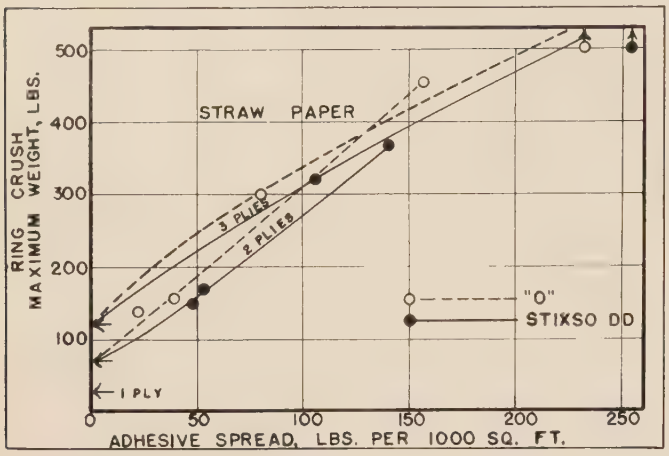

Fig. 1. Ring crush test on straw paper

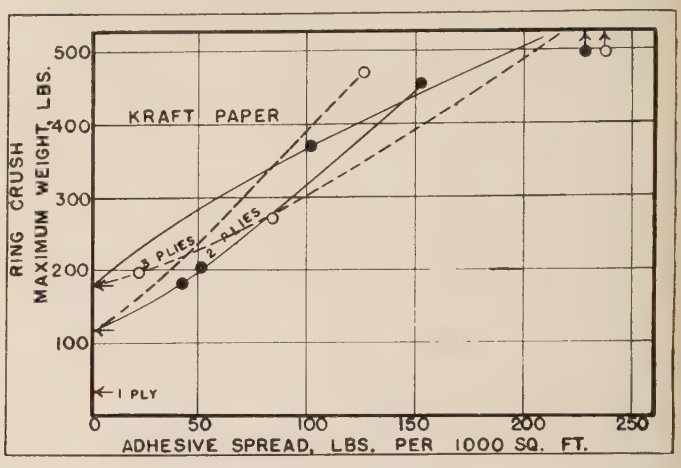

Fig. 2. Ring crush test on kraft paper

The adhesive was applied to small samples of paper by hand. A fairly uniform spread was obtained by sweeping an excess of adhesive across the paper with a glass rod wrapped at intervals with wire of known thickness to give the proper clearance. For medium spreads the clearance was 0.010 inch; for heavy spreads it was 0.035 inch. Light spreads were obtained by using no wire on the rod. Some three-ply samples were made by immersing the center ply in the adhesive and evacuating the air so that the silicate would penetrate better into the pores. These gave the heaviest spreads of all.

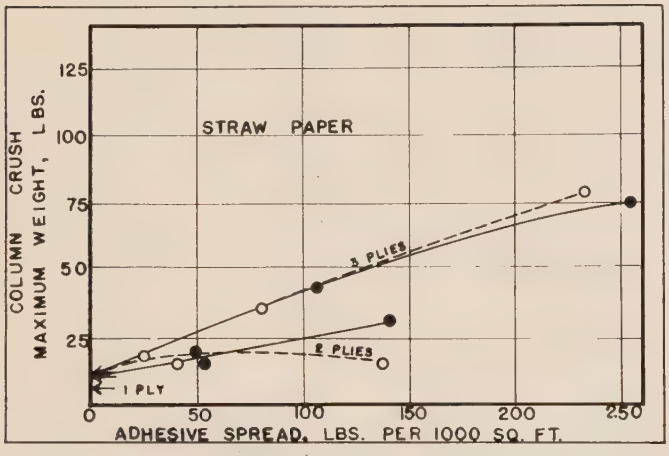

Fig. 3. Column crush test on straw paper

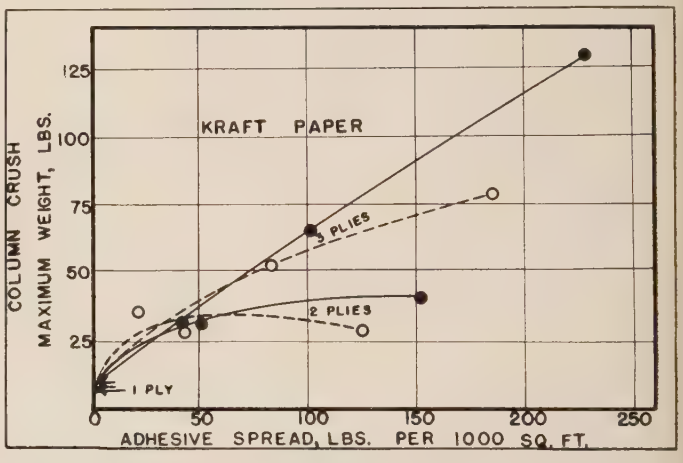

Fig. 4. Column crush test on kraft paper

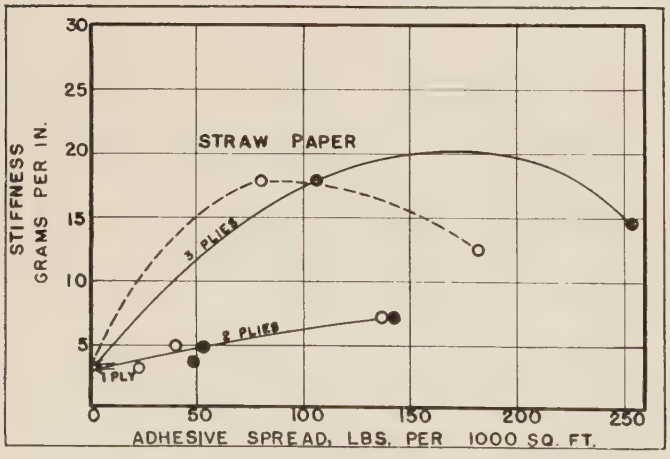

Fig. 5. Stiffness of straw paper

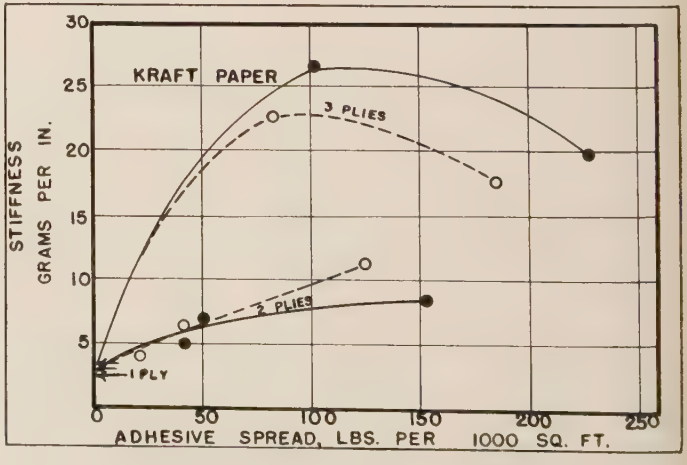

Fig. 6. Stiffness of kraft paper

After the adhesive had been applied the sheets were combined and held flat under weights until dry. They were then allowed to remain at a constant temperature of about 72°F. and a relative humidity of 50% until tested. In most cases the samples stood for 4 months, but those with light spreads stood for only 2 weeks. Just before testing, the paper was trimmed to the desired size.

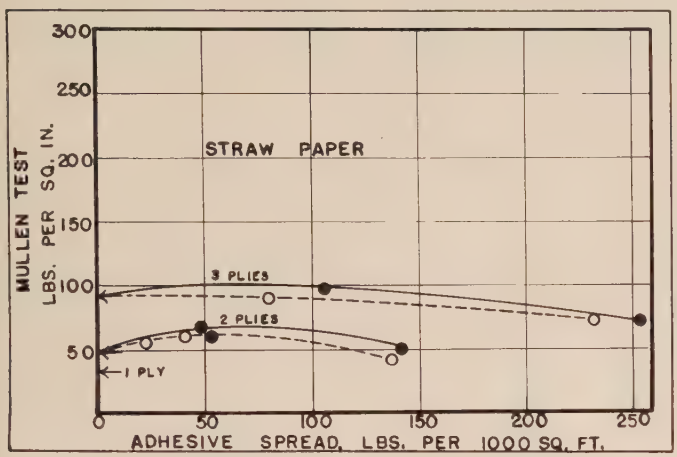

Fig. 7. Bursting strength test on straw paper

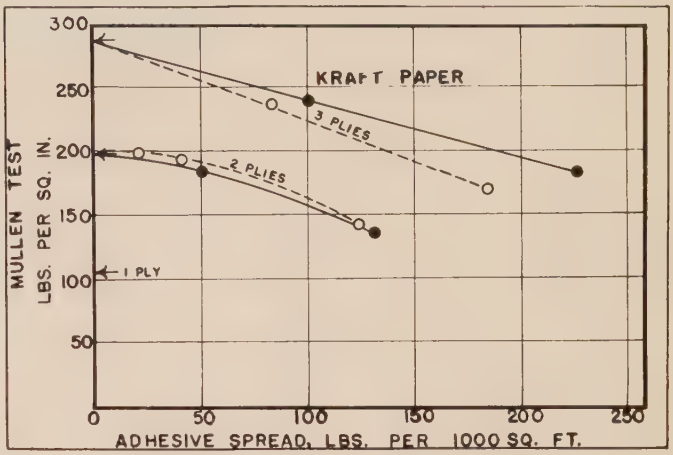

Fig. 8. Bursting strength test on kraft paper

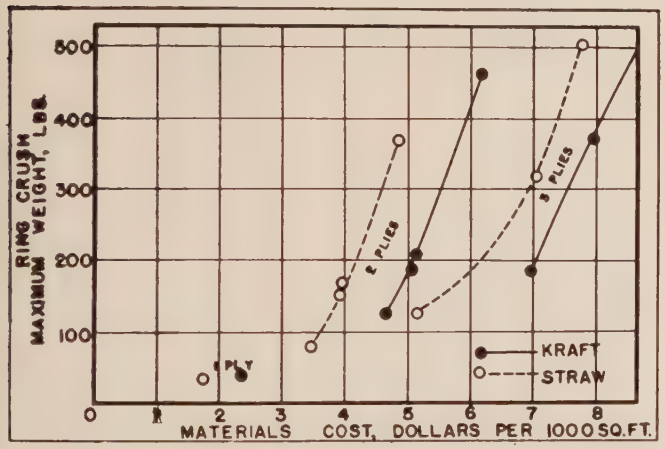

Fig. 9. Dependence of ring crush strength upon materials cost

The spread of silicate was determined by igniting samples of known area and weighing the ash. The spread was then calculated as pounds of liquid silicate per thousand square feet. The caliper and weight were also measured.

Single, double, and triple plies of unglued paper and double and triple plies with the various spreads of adhesive were studied. Each test was repeated three times with each combination. The tests were all carried out at 72°F. and 50% relative humidity.

The burst test was made in the usual manner using a hand-operated Jumbo tester with a 0 to 250-pound range. This test was included since it is standard and well known, even though it has little correlation with the strength of boxes (*2*).

The stiffness was determined by measuring the sag of a strip of paper supported at the ends and weighted at the center. A strip 10 by $^3/_4$ inch was suspended loosely at both ends and the sag was produced by hanging a 10-gram weight from a stirrup in the middle. The stiffness is listed in grams per inch of sag.

The other tests were made using a Thwing-Albert electrohydraulic tensile-strength tester, Model 35-4 (*1*). This instrument contains two plates which may be forced together at a constant rate. The force at which failure of the specimen occurs is obtained automatically. The maximum force that can be measured is 500 pounds. In these tests the plates approached at a constant speed of $3^1/_2$ inches per minute. Using this equipment, ring crush tests were made with strips $6^1/_4$ by $^1/_2$ inch looped to form a ring and laid on edge. The column crush test used a flat 2 by $^1/_2$-inch strip set on edge with the longer dimension horizontal. The force necessary to collapse the paper was determined. In a few cases with a heavy spread of adhesive the ring crush resistance exceeded the 500 pound capacity of the instrument.

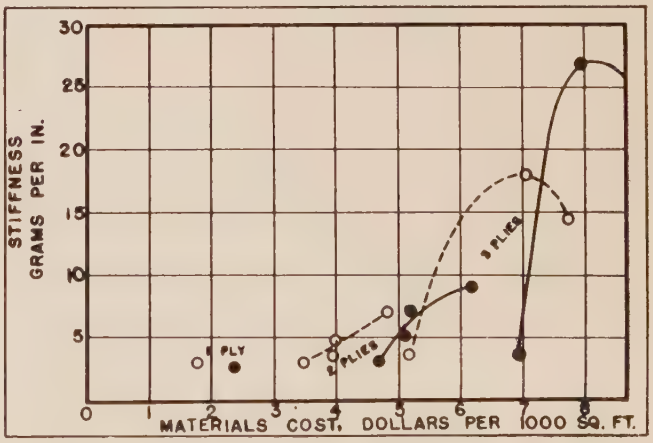

Fig. 10. Dependence of stiffness upon materials cost

RESULTS FOUND

The results are shown in Table II. Each figure represents the average of three trials. The spread of

adhesive as determined by ignition and the combined weight of board and adhesive in pounds per thousand square feet as well as its caliper are also included. The strengths are shown graphically in Figs. 1 to 8. Loose plies with no adhesive are indicated by arrows. Lines join the points for two and for three loose plies with the points for boards formed from these plies by application of various spreads of adhesive. The broken lines indicate board made with O silicate and the solid lines those made with Stixso DD.

In the tests involving rigidity the samples with silicate adhesive show a marked increase in strength over loose plies. This improvement is greatest with the ring test. In the column crush three plies show a much greater increase than two. In stiffness an optimum is found for three-ply board above which the stiffness decreases with increasing spread. With two plies a steady, but smaller increase occurs. With straw paper, the burst is little affected by the adhesive, but with the kraft the bursting strength is decreased appreciably.

These results show that sodium silicate contributes markedly to the strength of paperboard, often much more than an equal amount of paper.

Table II

Number plies	*Adhesive Type*	*Spread, lb./M sq.ft.*	*Total weight, lb./M sq.ft.*	*Caliper*	*Ring crush, lb.*	*Column crush, lb.*	*Stiffness, g./in.*	*Bursting strength*
				Straw paper				
1	..	0	38	10	35	7	3.0	35
2	..	0	76	20	77	10	3.0	52
3	..	0	114	30	124	11	3.6	94
2	O	23	85	22	156	19	3.1	57
2	O	40	107	24	157	16	4.8	61
2	O	137	164	27	453	16	7.0	42
2	DD	48	92	24	151	20	3.5	63
2	DD	53	108	25	168	16	4.7	61
2	DD	141	169	28	365	31	7.0	51
3	O	80	177	36	300	36	17.8	91
3	O	232	252	41	>500	79	12.3	73
3	DD	106	189	36	317	44	17.8	98
3	DD	254	261	39	>500	75	14.5	74
				Kraft paper				
1	..	0	44	10	37	8	2.7	110
2	..	0	87	20	123	9	3.1	202
3	..	0	131	30	184	10	3.6	292
2	O	21	100	21	200	37	4.1	201
2	O	42	113	23	186	29	6.4	178
2	O	125	163	27	474	29	11.4	145
2	DD	42	107	23	186	32	5.0	195
2	DD	51	115	24	206	31	7.0	168
2	DD	152	169	28	461	41	8.9	139
3	O	84	173	36	274	53	22.8	239
3	O	186	238	35	>500	79	17.8	177
3	DD	102	193	36	370	66	26.6	243
3	DD	228	240	36	>500	130	20.0	185

DISCUSSION

Because of the relatively low cost of sodium silicate, it should prove more economical to obtain a desired rigidity by using heavier applications of silicate rather than by using more plies or heavier paper. This is shown in Figs. 9 and 10 where the ring crush and stiffness are plotted against the cost of materials in dollars per thousand square feet. For the sake of brevity only Stixso DD is shown. Solid lines are drawn for kraft and broken lines for straw. The costs were based on prices of $105 per ton for kraft, $90 per ton for straw, and $1.00 per 100 pounds of silicate.

A hypothetical example is interesting for comparison. If a ring crush of 300 pounds were desired, it could be obtained with three 10-point plies of kraft and a 56-pound spread of silicate at a cost of $7.50. It could also be obtained with two plies of kraft and a 94-pound silicate spread for $5.60, or with two plies of straw and a 110-pound silicate spread for $4.55. The calipers would be about 33, 26, and 27, respectively, and the weights 165, 138, and 148 pounds per 1000 square feet.

Using some other test for stiffness as a criterion or working with another grade of paper or a different type of silicate adhesive would naturally result in somewhat different comparison, but it would be expected to be qualitatively the same as found here.

CONCLUSIONS

For many purposes rigidity is essential in paperboard boxes.

Rigidity of solid fiber board, as determined by resistance to crushing or bending, is increased greatly by use of heavy applications of sodium silicate. Board stiffness rises to a maximum with increasing amounts of silicate and beyond that decreases. With heavy silicate spreads, the burst test decreases more sharply on kraft than on straw.

Board rigidity is increased much more by the silicate than by an equal weight of additional paper. On a cost basis, the effect in favor of silicate is marked.

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A Quantitative Phloroglucinol-HCL Test in the Evaluation of Lignin Preparations

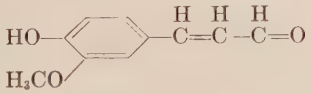
ROBERT M. DE BAUN and F. F. NORD

A quantitative determination of the color produced by lignins in reaction with phloroglucinol-HCl solution is described. The color reaction is interpreted in terms of β-hydroxy or α,β-unsaturated aldehydes. Samples tested include native lignins and lignins isolated by Soxhlet extraction, before and after decay by molds, from a typical softwood and several hardwoods.

ALTHOUGH it has been known (*1*) for many years that woody tissue gives rise to a red color when treated with acidified phloroglucinol solution, and although this reaction is supposedly due to the lignin in wood, so far no lignin preparation, excepting native lignin (*2*), gives this test. Native lignin appears to have the best chance of representing the lignin of the wood as it exists *in situ*. This is due to the fact that it is extracted by chemically inert solvents at room temperature. It has also been shown (*3, 4*) to be identical with a large portion of the residual, nonextractable lignin, as obtained by the cellulolytic action of wood-destroying fungi of the "brown-rot" type (4a).

Since native lignin gives this reaction we have applied a quantitative modification of this test to the evaluation of various lignins. This test has already been used in the study of some lignins obtained from cork (*5*).

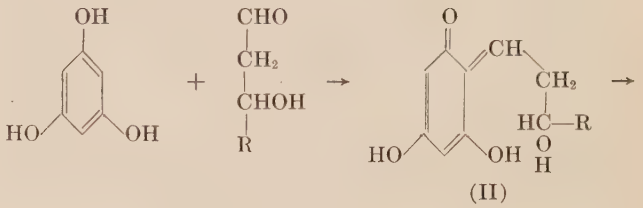
The first worker to attribute the phloroglucinol-HCl test to some specific component of wood was Czapek (*6a, b*) who obtained the so-called "hadromal." His work was criticized by Grafe (*7*), but was supported by Hoffmeister (*8*). However, these experiments were questioned by Pauly and Feuerstein (*9*). Crocker (*10*) thought that the wood reaction was due to traces of aldehydic substances such as coniferyl aldehyde which he thought the same as Czapek's "hadromal." Klason (*11*) was the first to implicate coniferyl aldehyde in lignin. Adler, et al (*12–14*) have shown that lignin preparations (lignosulphonic acid and native lignin) contain coniferyl aldehyde groups, and that the spectrum of the lignin-phloroglucinol-HCl complex is similar to those obtained with coniferyl aldehyde and its methyl ether. It is thus inferred that the coniferyl aldehyde group is responsible for the phloroglucinol-HCl test.



(I)

Coniferyl Aldehyde

Wenzel (*15*) attributed the red color produced with phloroglucinol and β-hydroxy aldehydes to a reaction of the following type.



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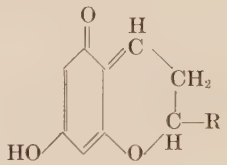
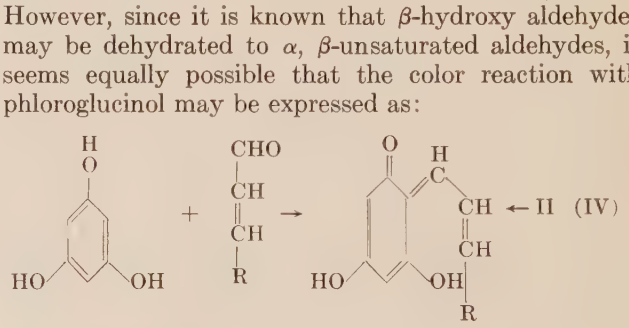


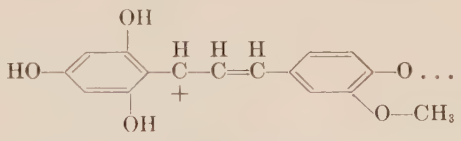
(III)

v. Wacek and Kratzl (*16*) have supported the "aldol" type of structure for the side chains of lignin, which would fit in with the formulation of Wenzel, above. However, since it is known that β-hydroxy aldehydes may be dehydrated to α, β-unsaturated aldehydes, it seems equally possible that the color reaction with phloroglucinol may be expressed as:



(IV)

The possibility of phloroglucinol reacting in a "keto" form is supported by the greater inherent stability of the keto over the enol form which would counteract the stabilizing influence of the resonance possibilities prevailing in the aromatic form (*17*). However, Ismay (*18*) has shown that even phloroglucinol trimethyl ether gives a red color with acid and wood. Thus it seems that the formulation (V) of Pew (*19*) is more satisfactory.



(V)

PROCEDURE

Five milliliters of the dioxan solution of the lignin to be tested, containing about 100 μ per ml. are taken in a colorimeter test tube. To this is added 1 ml. of concentrated HCl. To a separate, otherwise identical colorimeter test tube, 1 ml. of distilled water is added to 5 ml. of the same lignin solution. Three milliliters of phloroglucinol solution (0.1 *M* in 95% ethanol)

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are added to each tube rapidly with thorough mixing. This order of mixing permits the formation of the color to proceed at a more measurable rate, as already observed by Ismay (*18*). The blank is used to set the

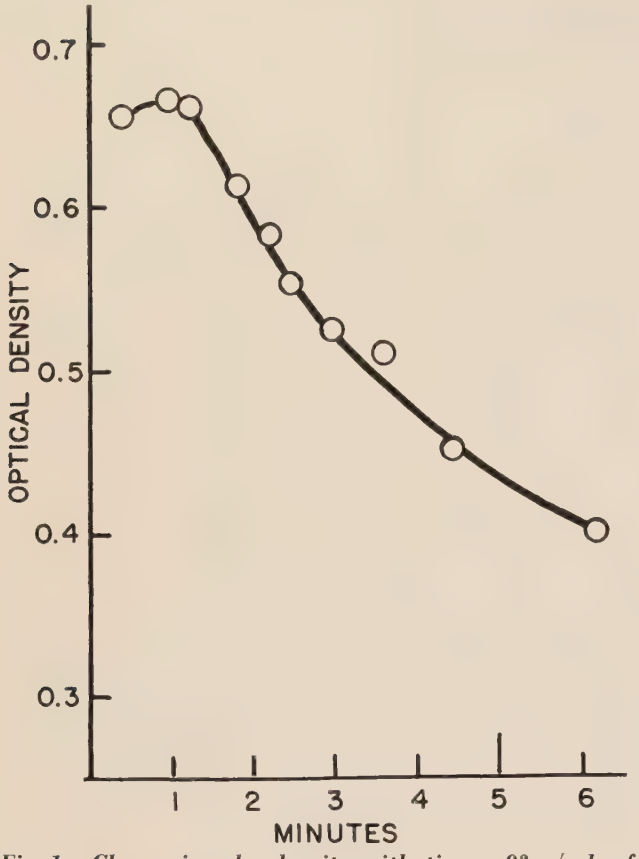

Fig. 1. Change in color density with time. 93 γ/ml. of white scots pine native lignin

colorimeter at zero density, and the color intensity is read after exactly 1 minute using a 550-mμ filter. In this case, a sample of white Scots pine native lignin was used as the standard.

Table I. Color Reaction with Several Lignins

Species and type of lignin	*Color reaction, %*			
White scots pine native (1)	100.0	101.0	101.5	100.0
White scots pine native (2)	107.5	108.0	108.0	...
White scots pine native (3)	122.5	125.0	...	...
White scots pine Soxhlet	113.5	110.0	109.0	...
White scots pine liberated[a]	89.6	90.0	88.0	...
Maple soxhlet (1)	55.2	55.5	54.0	54.3
Maple soxhlet (2)	47.7	47.8	48.0	...
Birch soxhlet (1)	9.5	9.0	9.0	...
Birch soxhlet (2)	19.4	20.1	...	...
Birch soxhlet (3)	17.1	19.6	...	...
Oak soxhlet (1)	41.2	43.0	...	...
Oak soxhlet (2)	46.5	47.0	...	...
Oak native (1)	7.0	6.0	8.0	...
Oak native (2)	21.4	21.9	...	...
Oak native (3)	49.2	47.0	...	...

[a] Native lignin, liberated by the cellulolytic action of *Lentinus lepideus*, partially purified.

The color reaction is quite sensitive to time (Figs. 1 and 2). A 550-mμ filter was used for the data in Fig. 2. The equation for the line (Fig. 3) was determined by the method of averages. Each point was evaluated in terms of this equation and the average deviation was found to be ±2.1 γ per ml. within the limits 0.0 to 5.0 γ per ml. in the range of 20 to 120 γ per

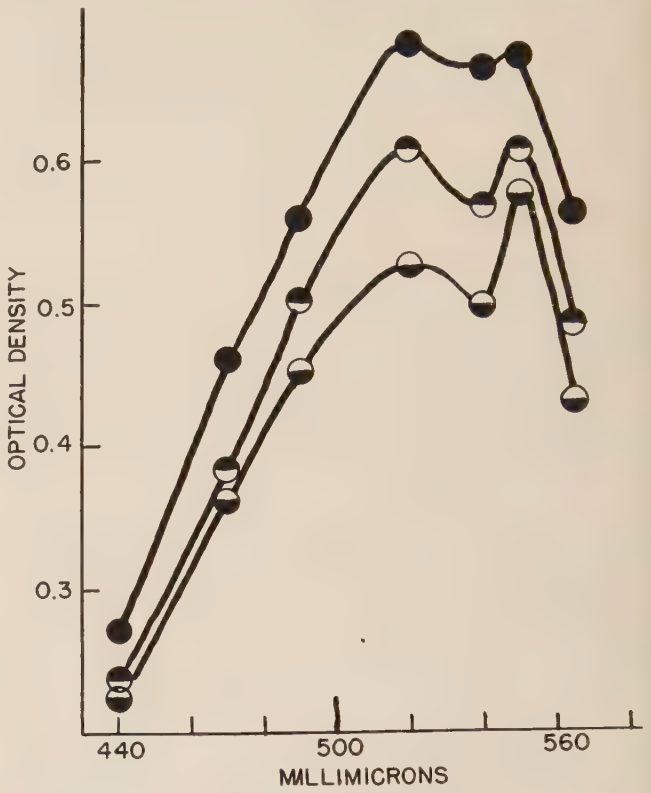

Fig. 2. Spectrum of the lignin color reaction: ● = 1 min.; ◓ = 2 min.; ◒ = 3 min.

ml. An Evelyn photoelectric colorimeter was used.

RESULTS

Replicate tests were carried out on several isolated lignin preparations, and the results are recorded in the following table.

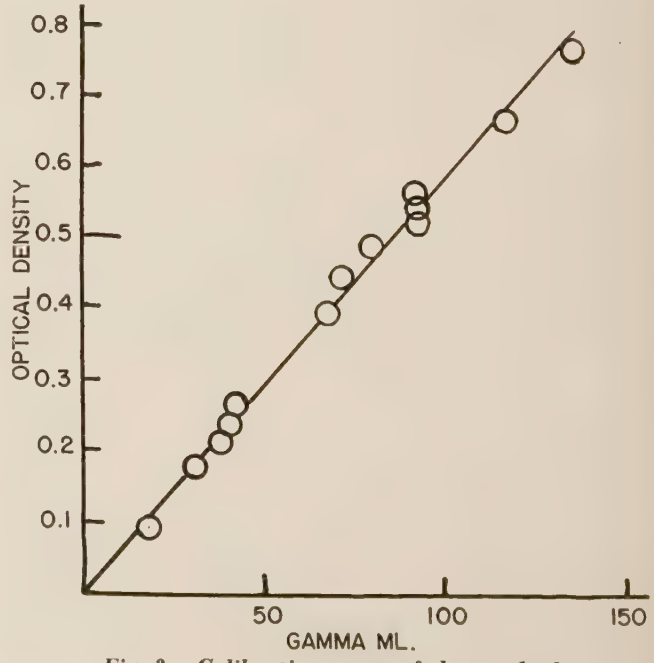

Fig. 3. Calibration curve of the method

DISCUSSION

Although the results are expressed in terms of the percentages of color reaction, what is really shown is the relative amount of chromogenic group in each preparation, which is probably the coniferyl aldehyde group. Thus what has probably been achieved is a kind of end-group analysis. It may be that these results are indicative of the degree of polymerization or of the molecular weight, but such an interpretation awaits the application of some other method which will give the particle size or weight of these lignins.

The similarity of the various lignins of white Scots pine is apparent as is the fact that the hardwood lignins (*20*) show less than one half the chromogenicity of the softwood lignin at this wavelength.

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Handling of Finished Pulp and Paper Products from the Machine to the Shipping Dock

H. F. R. WEBER

Many machines and devices are available for solving the problems of handling finished products. There are overhead cranes, monorails, trucks, tiering and manipulating units, gravity roller conveyors, powered conveyors, elevators, lowerers, and many others. For purposes of this paper only, a few are discussed, these being roll elevators and lowerers, pallet-type conveyors, both flush and elevated—these for handling rolls of finished paper, together with apron conveyors for handling bales of pulp, and trolley conveyors for handling trucks in a warehouse. The examples noted cover but a few of the types available for handling finished pulp and paper products. Each installation has been developed to solve a specific problem having as the final objective in each case the reduction of handling cost.

Within the lifetime of most of us, we have seen the use of paper expand from a few specific items to the point where it touches almost every phase of our lives. We have seen annual production increase from slightly more than 11 million tons in 1938 to 22 million tons in 1948. In spite of superb advertising by the industry, and a general public acceptance of paper products, competition is growing keen in certain lines and will be tougher after new facilities now under construction are in full production. This situation must be met with lower production costs if profit margins are to be maintained.

Production has been defined as materials in motion. There are two motions involved—processing motion and handling motion. Processing motion adds value to the product; handling motion simply adds cost. The greater part of indirect labor in most industries is engaged in moving and handling of materials, and in many industries a surprising amount of, what we consider as direct labor, is engaged in a similar purpose. Few manufacturers realize or appreciate the tremendous amount of time and money used daily merely to move material, or to use the generally accepted term—"materials handling." In foundries, for instance, it has been determined that from 150 to 200 tons of material are handled to produce 1 ton of castings. This may appear to be an extreme example, but we have seen figures for agricultural implement manufacturing which indicates that 180 tons of material are handled for each ton of finished product. It has been suggested that a fair average ratio for all manufacturing industry would be at least 50 to 1. Unfortunately, a similar estimate for the paper industry is not available, but we all know an over-all figure would be substantial.

Sometimes it is profitable to look at ourselves as others see us. Upon their return from a study of American plants recently, a British "productivity team" stated in their report, "In America materials handling is regarded as a major factor in the cost of living. Highly skilled attention is given to the movement of materials and thus has led to more efficient production techniques." Then, they went on to list the advantages of modern methods of handling materials. Among them were: reduced indirect labor costs, increased capacity of existing buildings, increased turnover, reduction of fatigue, conservation of skilled production labor, reduced cost of transportation, greater industrial safety, and improved outlook of workers operating mechanized equipment instead of doing hard manual labor.

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These advantages apply to the materials handling problems involved in every step of paper production—from forest to shipping room. The necessity for handling finished pulp and paper products warrants especially careful study. Since they have been completely processed, additional handling motions do not increase the value of these products but only increase their cost. Where such movements are necessary, properly designed and applied materials handling equipment can reduce those costs.

Many machines and devices are available for solving the problems of handling finished materials. There are overhead cranes, monorails, trucks, tiering and manipulating units, gravity roller conveyors, powered conveyors, elevators, lowerers, and many others. Each has its field of usefulness, but the author will limit his discussion to examples of powered conveyors, elevators, and lowerers with figures to illustrate specific applications in existing plants.

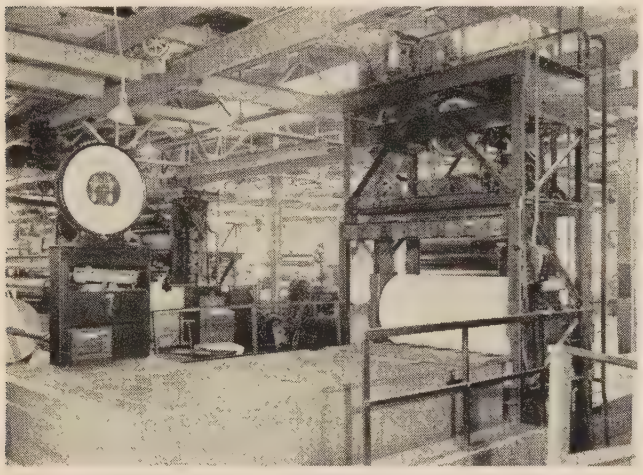

Fig. 1. Head end of lowerer with loading ramp. A similar ramp is located about 28 inches below in calender room on the opposite side of machine

At one plant (Fig. 1) rolls of high-grade paper up to 72 inches long are transported from paper machine and calender rooms to the floor below, where they are weighed, wrapped, headed, and stored before shipment. The rolls are conveyed between floors without damage, quickly, economically, and safely by a motor-operated, skip-type lowerer. Equipped with a single finger-type tray, this device occupies a minimum of floor space and is suitable for any reasonable height.

Figure 1 illustrates the head end of the lowerer with its loading ramp in the paper machine room. A similar ramp is located about 28 feet below in the calender room on the opposite side of machine.

The scale shown at left records net weight, and the dolly is used to center the rolls before they are delivered to the lowerer tray. A lift gate is raised and lowered automatically when handling rolls from this side, or its rubber padded bumper may be used for accumulating rolls when the machine is set for loading from the opposite side.

The drive machinery is located up out of the way, and the supporting structure occupies a minimum of floor space.

The foot end of lowering machine is shown in the background of Fig. 2. The roll is resting on the finger-type lowering tray just before being gently discharged to the ramp by rubber-faced unloading fingers. The rolls are turned 90° on the turntable in the foreground, centered on the transverse dolly, and rolled beyond to the left for the wrapping and heading operations. Also, at the left side of the figure may be seen the drive end of the storage conveyor which is shown in Fig. 3.

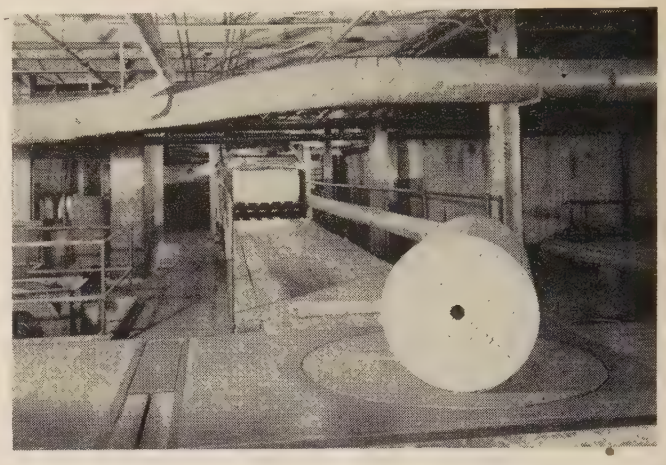

Fig. 2. Foot end of lowering machine with roll resting on finger-type lowering tray ready to be discharged. Notice turntable in foreground where rolls are turned 90° and directed to the wrapping and heading operations

After being wrapped in the area shown in the right background (Fig. 3), the rolls are again weighed and delivered to this storage conveyor. From it they are rolled to storage tables on the left, from the opposite end of which they are removed for shipment.

The conveyor is 130 feet long, made up of a series of V-top cars, connected and propelled by a single chain. It is reversible and operates at 60 f.p.m. Push buttons are located at various points along the line. The drive end of the storage conveyor is equipped with a safety switch to prevent over travel of rolls.

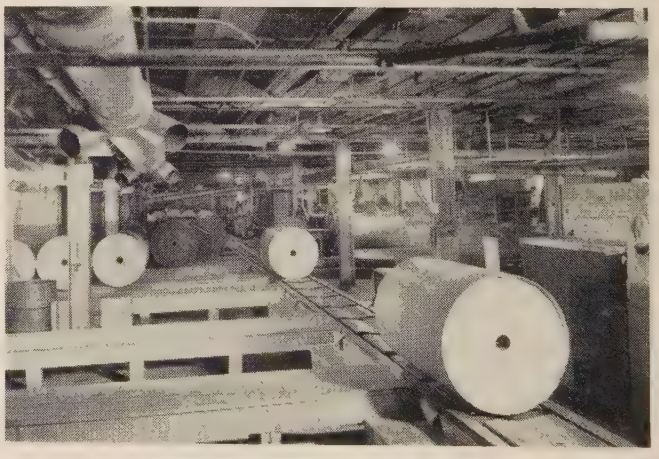

Fig. 3. Storage conveyor made up of a series of V-top cars connected and propelled by a single chain. It is reversible and operates at 60 f.p.m.

The whole system is simple, safe, and reliable. It has been the means of handling, wrapping, heading, weighing, and storing rolls efficiently within a minimum of plant floor space.

The skip tray-type unit in another plant is somewhat similar to the one described previously, except that it is designed to elevate as well as lower the rolls between floors.

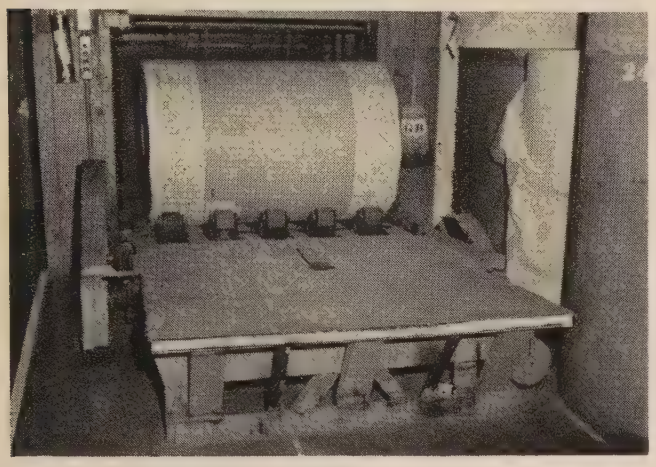

Fig. 4. Cradle has rotated sufficiently to start pushing the roll into the elevator carrying tray. Note also roller mounted loader arms are in a position to retard next roll when placed on the platform

In still another plant, the roll elevator consists of a single tray suspended on two chains. It operates in guides, which are arranged to tilt the tray to discharge rolls automatically on the floor above.

The loading station is equipped with a motor-driven, tilting, cradle-type loader. It is interlocked electrically with elevator, and the roller-equipped cradle arms are inclined to retard the roll until the hoisting tray is in place.

In Fig. 4 the cradle has rotated sufficiently to start pushing the roll into elevator carrying tray. At the same time, the roller-mounted loader arms are in a position to retard the next roll when it is placed on the platform.

Figure 5 illustrates the elevator discharging rolls on the upper floor. Note the safety door which is automatically closed as elevator descends.

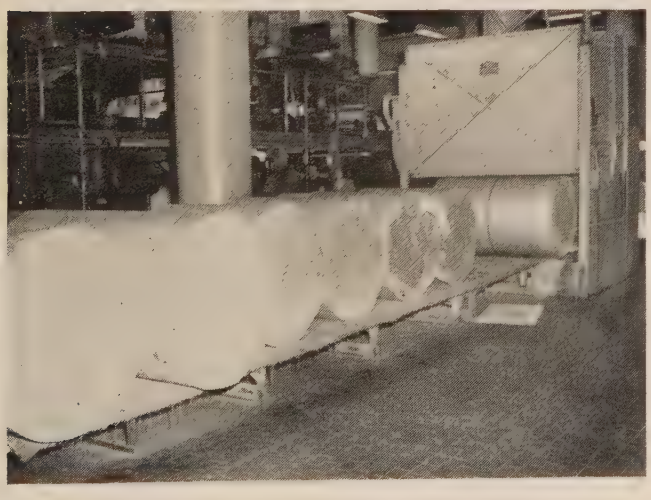

Fig. 5. Elevator discharging rolls on the upper floor. Note the safety door, which is automatically closed as the elevator descends

This elevator is quite simple, requires very little power for gently handling single rolls up to 5000 pounds at hoisting speeds of 75 f.p.m.

A similar installation has been in successful operation for 15 years in a large newspaper plant. In this particular case, the rolls are transported by gravity on dollies through tunnels under the city streets from a storage warehouse to the elevator, in which they are elevated 44 feet to the press room floor. The elevator consists of a series of trays suspended from two strands of chain, and is designed to handle 36-inch diameter, 2000-pound rolls at the rate of two per minute.

The scope of usefulness of elevators of this kind is very broad. They can be made to handle rolls of highly finished paper very gently and just about as automatically as conditions require. There is virtually no limit to their height, and they can be arranged to receive and discharge selectively at several floors with capacities up to 2 or 3 per minute. They provide maximum safety for personnel and reduce manual handling to a minimum.

Previously, the author described an application of a pallet conveyor serving the wrapping and heading department of a large paper mill. Figure 6 illustrates a somewhat similar conveyor which serves as the means of transporting rolls along one side of the warehouse and shipping floor of another mill. It is 14 inches wide by 160 feet long and handles rolls up to 42 inches in diameter by 80 inches long, weighing up to 4000 pounds

Fig. 6. View of conveyor showing open space around conveyor. Note also conveyor can be used for live storage.

each. The railroad loading track extends along the far side of the room, permitting rolling transfers directly from warehouse floor to box cars.

A similar heavy-duty pallet conveyor, also mounted flush with the floor, serves the calendering room on the floor above.

Incidentally, in the upper part of the slide is shown an overhead flat belt conveyor suspended from the ceiling for handling "broke" from the calendering room floor directly into the "broke" beater in the beater room.

Figure 6 shows clearly how the rolls can be loaded or unloaded at any point in the path of the conveyor. The conveyor can also be used for "live" storage and inasmuch as conveyor is flush with the floor, additional beams, etc., usually included in conveyor systems, are not necessary. This contributes to better housekeeping and easier maintenance.

An economical method of handling finished stocks of paper in a warehouse is clearly illustrated in Fig. 7.

This particular warehouse contains 12,000 square feet of office space, 162,000 square feet of warehouse space, and has 30,000 square feet of dock space. It serves a wide area with orders ranging from a ream to a car load.

Fig. 7. View of the conveyor showing two long parallel runs. Conveyor is so arranged that drive cuts out automatically in case of fire

An overhead trolley conveyor, 1200 feet long, is used to tow trucks of merchandise from various sections of the building to the shipping dock. These trucks weigh 300 pounds empty and up to 2300 pounds loaded.

Sales orders are dispatched by a pneumatic system from central office in the shipping room to the department or warehouse area from which required items may be obtained. A man in the department receiving such an order detaches an empty truck from the conveyor loads onto it the required items, marks the destination of the material on a board mounted on the telescopic mast of the truck, and attaches the truck to the conveyor.

The whole procedure is simple and economical, and provides a most flexible method of assembling a wide diversity of orders quickly.

The conveyor (Fig. 7) has long horizontal runs, winding around a number of roller turns and passes through several fire doors enroute. It is propelled at a speed of 100 f.p.m. by a booster drive equipped with a 5-h.p. electrofluid drive which assures smooth starting under all conditions and minimum power consumption. Furthermore, this drive will cut itself out automatically in case of a fire, resulting in fire doors closing down on the conveyor.

This one trolley-type truck tow conveyor performs all of the functions of an inter-department transportation system—and does the job economically, safely, and dependably.

Because so many mills must supplement their own production with purchased pulp, it was felt that handling baled pulp could well be included in this paper.

In Fig. 8 is shown the inclined portion of a 36-inch wide reversing steel apron conveyor for handling 400-pound bales of sheet pulp from box car unloading platform direct to beaters, or to storage at end of horizontal run and later returning them to beaters as needed, when the conveyor is operated in reverse.

The bales are delivered directly to the inclined foot of the conveyor from a hand truck, the railroad truck being directly adjacent with the floor of the box cars at the same elevation as the unloading platform.

When handling bales of pulp from car to storage, the bales travel to a stop located at the head end of the conveyor in the warehouse. Since the conveyor has a smooth, flat surface, it easily continues its travel bringing up several more bales, placing them tightly together to permit removal en masse by an overhead crane sling to pile them high alongside the conveyor or in some other place in the warehouse. Maximum speed of this conveyor is 60 f.p.m.

The examples discussed illustrate but a few of the types of equipment suitable for handling finished pulp and paper products. Each installation shown was developed to solve a specific problem, having as the final objective in each case the reduction of handling costs. Other types of equipment and various arrangements are available, and have been used successfully to meet the particular requirements of other plants. Success with installations of this kind depends on equipment that is engineered to meet specific conditions.

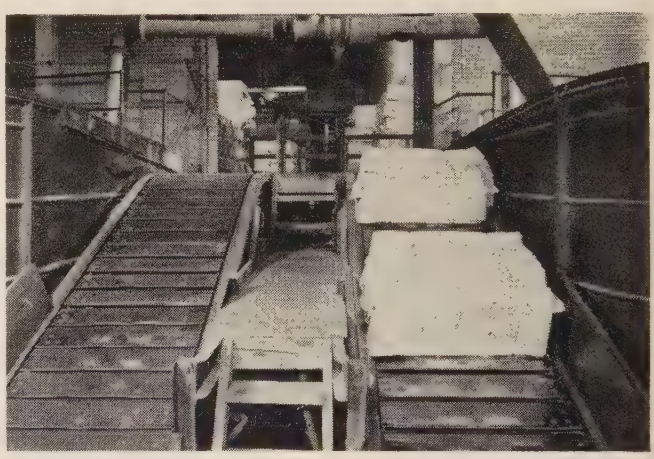

Fig. 8. Inclined portion of reversing steel apron conveyor for handling bales of sheet pulp from unloading platform to beaters or to storage

Often, the possibilities for cost reduction by the use of materials handling equipment are obscured by familiarity with, and unchallenged acceptance of, customary methods or operations. For that reason, most mills could profit from a thorough study of their processes and intra-plant transportation of materials in order to determine where potential savings are possible. In making studies of this kind, do not overlook the ideas of the men in the mill. Much of the progress that already has been made is the result of ideas conceived and put into effect by operating personnel. The interest and cooperation of the front line operating employee, with his constant association with handling problems, is an essential part of any program designed for modernizing handling practices. Such studies will pay substantial dividends.

The conveyor industry is eager to cooperate with engineering and operating groups of the Paper Industry in meeting the challenge for lower handling costs and increased efficiency of your processes.

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A New Color Reaction of Cationic Soaps and Its Application to Studies of Their Interactions with Cellulose Fibers

JOHN A. HART and EDWARD W. LEE

A new color reaction of alkaline earth metal lakes of the dyestuff Eriochrome Azurol B with cationic soaps is described. The reaction was found to provide a very sensitive means of determining cationic soaps in dilutions as great as 1 part in 10 million. The adsorption of cationic soaps on cellulose fibers of varying degree of purity and mechanical refinement has been measured, and the data are discussed. The dyestuff is shown to serve as a good indicator in the determination of anionic soaps by titration with cationic soaps.

When using Eriochrome Azurol* to color cellulose ester films it was noticed that the dye solutions and the colored films showed differences in color during a period of aging, which were then assumed to result from changes in the state of aggregation of the dyestuff molecules. On this supposition it seemed reasonable to expect that comparable changes might be induced by adding surface-active compounds which by reason of their cationic nature would tend to become oriented about the polar regions of the dyestuff molecule. It was found that alkaline solutions of the dye in the presence of some metals, notably calcium and magnesium, were very sensitive to cationic soaps, but it now seems improbable that the theoretical assumptions which led to this discovery were wholly correct.

The dyestuff dissolved in dilute aqueous ammonium hydroxide to a yellow solution which changed to a brilliant purple when calcium or magnesium salts were added. This color change may be of value for the determination of these metals. With calcium, the purple color changed through blue to green when a cationic soap was added; with magnesium, a further change to yellow took place.

CATIONIC-ANIONIC SOAP TITRATIONS

The method of Hartley and Runnicles (*1*) for the determination of anionic soaps by titration with pure cetyl trimethylammonium bromide, using bromophenol blue as indicator, is simple and convenient. Pankhurst and Smith (*2*) have used the method effectively in their studies of the interactions of sodium dodecyl sulphate and gelatin, and have described the lighting conditions most suitable for observing the end point of the titration. The end point in this titration, a change from the normal alkaline purple of bromophenol blue to a pure blue in the presence of an excess of cetyl trimethylammonium bromide, was quite sharp when sodium dodecyl sulphate was titrated; in a titration requiring about 25 ml. of $N/1000$ cetyl trimethylammonium bromide to reach the end point, the uncertainty was about 0.2 ml. When titrating some other anionic soaps, such as the commercial wetting agent Teepol X,* the color change extended over about 1 ml. of the standard solution. Further difficulty in observing the end point was experienced with those anionic soaps which form insoluble complexes with cetyl trimethylammonium bromide, when the titrated solution became progressively more turbid during the titration. Other anionic soaps of commercial purity had a brownish or yellow color which tended to obscure the end point. It was expected that Eriochrome Azurol would be a better indicator in this titration because of the more brilliant color changes it showed.

For use as a titration indicator, the dyestuff was prepared as a 0.3% solution in alcohol. A few drops of this solution were added to 25 ml. of the anionic soap solution to be titrated, followed by 5 ml. of ammonium hydroxide solution (S.G. 0.920). The tap water used to prepare the anionic soap solutions contained sufficient calcium, so that no addition was necessary.

The color change at the end point was more pronounced than that of bromophenol blue and could be seen in either tungsten or daylight. Turbidity and color in the titrated solutions did not obscure the end point so greatly as with bromophenol blue.

Sharpness of end point depended, as with bromophenol blue, upon the nature of the anionic soap being titrated. Broad end points were also found when cetyl trimethylammonium bromide was replaced by cetyl ethyl dimethylammonium bromide, even in the case of these anionic soaps which gave sharp end points with cetyl trimethylammonium bromide. Differences in the sensitivity of the indicator in these titrations may be analogous to the differing sensitivities of indicators in acid-base titrations according to the relative strengths of the reacting acids and bases. A property of surface-active substances in solution depending upon the relation of polar and nonpolar molecular substituents and the character and degree of ionization of the polar portion may be analogous to hydrogen ion concentration in solutions of simple acids and bases.

The reverse titration, i.e., the determination of cationic soaps by titration with a standardized anionic soap solution was not satisfactory because the indicator color change was considerably less clear; it was not fully reversible, and was slower.

COLORIMETRIC DETERMINATION OF CATIONIC SOAPS

For the determination of cationic soaps a colorimetric procedure based on the color changes of the magne-

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* Color Index No. 720. The condensation product of ortho cresotinic acid and ortho dichlorobenzaldehyde, Geigy Co.
* A sulphated petroleum fraction. Shell Chemicals Ltd.

sium lake of Eriochrome Azurol B was developed. It proved convenient and capable of high sensitivity.

A magnesium dye-lake dispersion was prepared by mixing two solutions immediately before use. It could not be prepared as a single solution because the magnesium-dye complex tended to separate as a brilliant blue film at the air-water interface, which formed into tiny flocs when ruptured by shaking or pouring the solution. The quantity of dye lost in this way from the dispersion was small, but the flocs accumulated after the stock solution had been used a few times and tended to interfere with optical measurements.

The procedure in determinations of cationic soaps was the following:

Dye solution		
Eriochrome Azurol B	0.4	grams
Potassium carbonate (anhyd.)	27	grams
Water to	2000	ml.
Magnesium solution		
Magnesium sulphate	25	grams
Polyvinyl alcohol*	10	grams
Water to	2000	ml.

* Shawinigan Chemicals Ltd. grade 95/50.

Ten milliliters of the dye solution and 10 ml. of the magnesium solution were mixed (the order of mixing was immaterial) and then 25 ml. of the solution to be determined were added. The presence of 1 part in 1 million of a surface-active quaternary ammonium salt in the latter solution was sufficient to cause a readily visible color change. When using a Klett photoelectric absorptiometer with a red filter, 1 part in 10 million was readily detectable.

The colors produced tended to be unstable, more especially when the quantity of cationic soap present was sufficient to produce a pure blue color. Over a period of a few hours the blue dispersion became opalescent and reddish in hue and finally flocculated. This change was inhibited by polyvinyl alcohol, and was then sufficiently slow as to cause no difficulty in the practical use of the method.

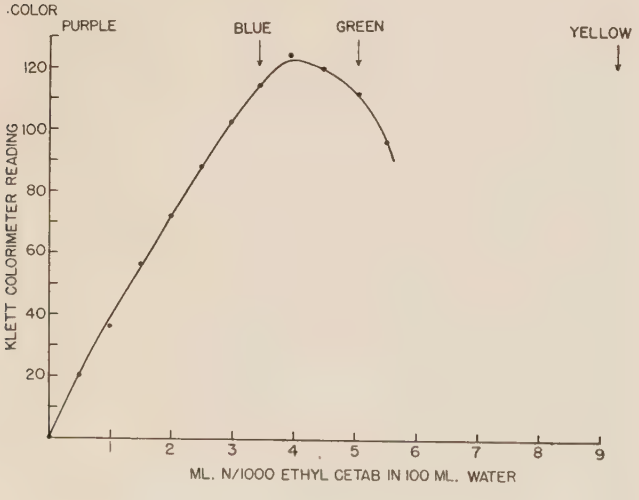

Fig. 1.

Measurements made with a Klett Absorptiometer for various concentrations of Ethyl Cetab (commercial cetyl ethyl dimethylammonium bromide)* are shown in Fig. 1. Higher concentrations than that which produced a pure blue color required the use of different light filters. At low concentrations the sensitivity of the Klett instrument was seriously limited by scattering caused by dust or smears on the sample tube surfaces. An instrument designed to measure diffuse light density rather than something approaching parallel light density would have been more suitable.

* Rhodes Chemical Corp.

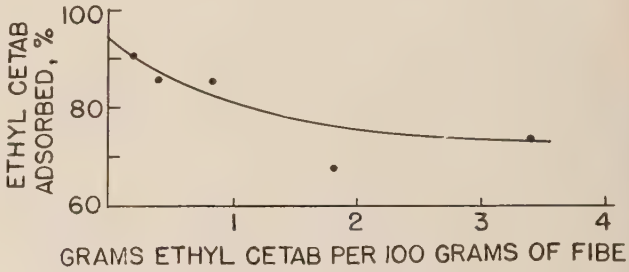

Fig. 2. Fiber in water concentration 0.4%

After each measurement the sample tube was rinsed with a dilute mineral-acid solution to remove the thin film of dyestuff adsorbed upon the glass surface. When tubes were washed with water alone, errors due to the optical density of this film became appreciable after the tubes had been used a few times.

The procedure was most effective when used to determine cationic soaps derived from strong bases, such as quaternary ammonium, pyridinium, and quinolinium hydroxides. Greater concentrations of the more weakly basic surface-active primary and secondary amines were required to bring about the color changes, but nevertheless the method is likely to be of value for determinations of these compounds also.

INTERACTION OF CATIONIC SOAPS WITH CELLULOSIC FIBERS

The interactions of cationic soaps with cellulose are of considerable theoretical interest, and in the fields of textile and paper technology they are of practical importance. Cationic soaps are used widely to confer a softer handle or a water-repellent surface upon textile fabrics, and have been suggested as softening agents for papers. It is well-known that these substances are exhausted from solution by cellulose fibers, as are dyestuffs, but this exhaustion is not complete. When cationic soaps are added to papermaking pulps before forming the paper, completeness of exhaustion is a factor of great economic importance. In a typical pulp suspension at the time of forming paper, with a cationic soap added to the extent of 1% of the pulp fiber weight, the fiber concentration is 0.2% and the cationic soap concentration only 0.002%. If 99% of the soap were fixed upon the fiber the concentration of soap remaining in the water would be 0.00002%, or 2 parts in 10 million. Determination of such low concentrations requires the use of a very sensitive analytical procedure.

Some studies of the interaction of cetyl ethyl dimethylammonium bromide with papermaking fibers have been made. Although these studies were not exhaustive, the results obtained were judged to be of sufficient interest to present here.

A typical bleached sulphite softwood pulp in the form of wet lap was disintegrated in a solution of Ethyl

Cetab in distilled water by means of a high-speed stirrer, and allowed to react for 3 hours, with frequent gentle stirring. At the end of this time the suspension was filtered, and the concentration of the cationic soap remaining in the filtrate was determined by the colorimetric procedure described.

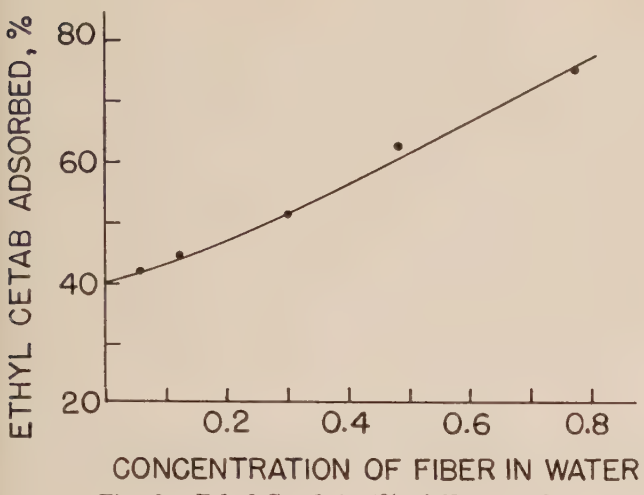

Fig. 3. Ethyl Cetab 3.75% of fiber weight

Proportions of pulp fiber, cationic soap, and water were varied independently over a wide range. The results are illustrated in Figs. 2, 3, and 4.

It would appear from these data that the cationic soap is not completely exhausted from solution, but that its interaction with the pulp fiber is of the nature of an adsorption process comparable with the process by which water vapor is adsorbed from air by cellulose.

Table I

	Catonic soap adsorbed, %
Bleached sulphite softwood pulp, sample No. 1, beaten 0 min.	62
Bleached sulphite softwood pulp, sample No. 1, beaten 5 min.	62
Bleached sulphite softwood pulp, sample No. 1 beaten 20 min.	62
Bleached sulphite softwood pulp, sample No. 1, beaten 35 min.	62
Bleached sulphite softwood pulp, sample No. 1, beaten 50 min.	62
Bleached sulphite softwood pulp, sample No. 2, beaten 0 min.	62
Bleached sulphite softwood pulp, sample No. 2, beaten 5 min.	62
Bleached sulphite softwood pulp, sample No. 2, beaten 20 min.	62
Bleached sulphite softwood pulp, sample No. 2, beaten 35 min.	62
Bleached sulphite softwood pulp, sample No. 2, beaten 50 min.	62
Bleached sulphite softwood pulp, sample No. 3	65
Bleached sulphite softwood pulp, sample No. 3, dried at 100°C	64
Unbleached sulphite softwood pulp, sample No. 1	98
Unbleached sulphite softwood pulp, sample No. 2	92
Unbleached softwood kraft pulp	97
Neutral sulphite semichemical hardwood pulp	95
Poplar mechanical pulp	98
Bleached soda pulp	78
Commercial pulp, described as "high alpha," sample No. 1	63
Commercial pulp, described as "high alpha," sample No. 2	55
Cotton linters	39
Cotton linters, beaten 35 min	42
Filter paper, sample No. 1	37
Filter paper, sample No. 2	37

Similar experiments were made to discover the behavior of different kinds of cellulose fiber. In each case the fiber-in-water concentration was 0.33% and the quantity of cetyl ethyl dimethylammonium bromide was 2% of the fiber weight. Because some dried fibers were included in the range studied, a longer reaction time—24 hours— was allowed.

The effect of beating was also examined. Beating was carried out in a Valley laboratory beater according to TAPPI standard procedure for pulp testing.

The results of these experiments are shown in Table I.

It is clear from these results that although the cationic soap was adsorbed to fibers of nearly pure cellulose, the greater part of the adsorption to commercial woodpulp fibers was due to another constituent, probably lignin. It is understandable that the phenolic hydroxyl groups of native lignin and the sulphonic acid groups of partly sulphonated residual lignin in unbleached sulphite pulps would have more affinity for basic ions than the feebly ionized hydroxyl groups of cellulose.

The absence of any substantial effect by beating pulp fibers suggests that the area of fiber surface exposed does not determine the degree of adsorption. This is consistent with the findings of other investigators that the water-binding capacity of cellulose is not changed appreciably by beating. It should be observed however that the action of the Valley beater used differs markedly from that of other beaters such as some kinds of ball mills and the stone-roll beaters used in greaseproof paper manufacture, in that it does not promote in so great a degree (for a given degree of fiber cutting) the change in pulp properties which gives strong, tough papers and which is known to papermakers as hydration or wetness.

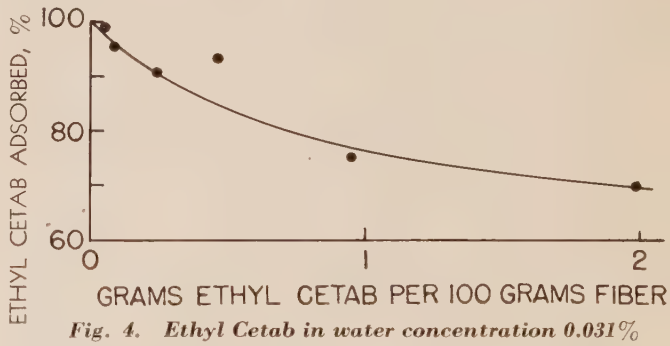

Fig. 4. Ethyl Cetab in water concentration 0.031%

Although estimates of the surface area of pulp fibers are of doubtful accuracy, it seems likely that the quantities of cationic soaps which are adsorbed to them are much in excess of that required to form a solid monolayer. It is probable that the adsorbed layer is of gaseous rather than solid film type.

The effects observed in these experiments are analogous to those observed when cellulose fibers are colored with basic dyestuffs.

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Levulinic Acid from Wood Cellulose

THOMAS R. FROST and E. F. KURTH

Levulinic acid was produced from wood cellulose with mineral acids. Major yield-determining factors were found to be temperature, time, catalyst, and concentration of catalyst. Investigations were carried out using temperatures from 150 to 190°C.; times from 1 to 3 hours; and catalysts that included hydrochloric acid, hydrobromic acid, sulphuric acid, and some salts. Acid catalyst concentrations were varied from 2 to 8%. The conditions for the production of the highest yield of levulinic acid from cellulose with hydrochloric acid were found to be quite flexible; i.e., at 150°C., the yield was 44.6% with 2% acid at the end of 3 hours, 46.24% with 4% acid at the end of 2 hours, and 45.91% with 6% acid at the end of 1 hour. At 170°C. the yield was 46.85% with 2% hydrochloric acid after heating for 1 hour. Hydrobromic acid gave the maximum yield of 56.25% at a 4% concentration and heating for 1 hour at 170°C. Sulphuric acid gave lower yields under all conditions. The presence of arsenic trichloride inhibited the formation of levulinic acid, zinc chloride was of no benefit, and stannous chloride improved the yield. Further, it was found that when wood was subjected to similar conditions, it also produced levulinic acid plus furfural.

THE purpose of this investigation was to explore another means for utilizing wood residues through the production of levulinic acid. It long has been known that hexose sugars treated with a mineral acid gave rise to formic and levulinic acids. Stoichiometrically the relationship between a hexose and levulinic acid is given by the traditional equation:

$$C_6H_{12}O_6 \rightarrow CH_3\,COCH_2\,CH_2\,COOH + HCOOH + H_2O$$

This relationship is not rigid, for various side reactions occur with the result that carbon dioxide and humin are also formed in considerable quantities.

Investigations on the production of levulinic acid from sugars (*2, 4, 11*), agricultural wastes (*9*), and starch (*3*) are reported in the chemical and patent literature. A yield of 72% is claimed from sucrose (*2*) and 48.5% from glucose (*4*) by heating with hydrobromic acid. One patent relates to the manufacture of the acid from pine wood (*7*). From the outset of this study, it was assumed that levulinic acid could be produced from cellulose and wood with fair yields, but the conditions, consistent with good economy, for obtaining the optimum yield were unknown.

Before an investigation into these conditions could be started, an acceptable method for the quantitative estimation of levulinic acid had to be selected. There were three possibilities open for a quantitative method, based on the three different functional groups of the levulinic acid molecule. These are the carboxyl, keto, and acetyl groups. The use of any of them has its drawbacks, but the method for the acetyl group was chosen as being best suited for the work at hand. The analytical procedure adopted stemmed from the work of Ploetz and Bartels (*5*), who elaborated on an earlier work by Savare (*6*). Savare based his investigation on the iodoform reaction and computed his results on the usual basis of 6 iodine atoms used per molecule of levulinic acid. Ploetz and Bartels, however, found this determination to be inaccurate, as the results were dependent on the amount of potassium hydroxide used in the reaction. By adding excess base, they found that known amounts of levulinic acid could be determined with an error of ±4%. Also, these investigators computed their results on a basis of 8 iodine atoms per molecule of levulinic acid. Further, they claimed that in the presence of the excess base, the 8-iodine reactions occurred to the exclusion of the 6-iodine reactions. This method seemed to meet the requirements of the present investigation.

EXPERIMENTAL WORK

In starting the investigation, the initial major factors to be resolved were two: namely, an apparatus adequate for the study and an examination of the analytical procedure to determine its reliability.

Apparatus

The requirements for a suitable apparatus called for an autoclave capable of being heated to temperatures of 150 to 190°C. in a relatively short period of time. Once at the desired temperature, the pressure should be maintained within rather close limits for the duration of the cook. It would be advantageous to rotate the autoclave to promote mixing while the reactions are in progress, and relief of pressure should be carried out in a short time. The autoclave preferably should be of such a size as to accommodate several sample bombs.

Of the equipment available, a small stainless-steel pulping digester was deemed most suitable. This machine had a working temperature range up to 205°C., and it was fitted so that it could be heated either by direct steam or by injecting steam into the digester jacket. The time to reach the desired pressure on experimental cooks was found to be less than 5 minutes, and relief could be accomplished in the same time interval. The steam line was equipped with a pneumatic thermostatic control valve for temperature regulation. Regulation could be checked by means of a recording thermometer, the thermocouple of which was located inside the digester cooking chamber. The digester was mounted on trunnions and could be rotated during the entire cooking cycle.

To hold the samples during the cook, 8-inch Pyrex, chemical-resistant glass, and soft glass test tubes were fashioned into bombs by sealing the open ends. Trial cooks were then made with actual samples—5 grams of cellulose in contact with 50 ml. of a mineral acid.

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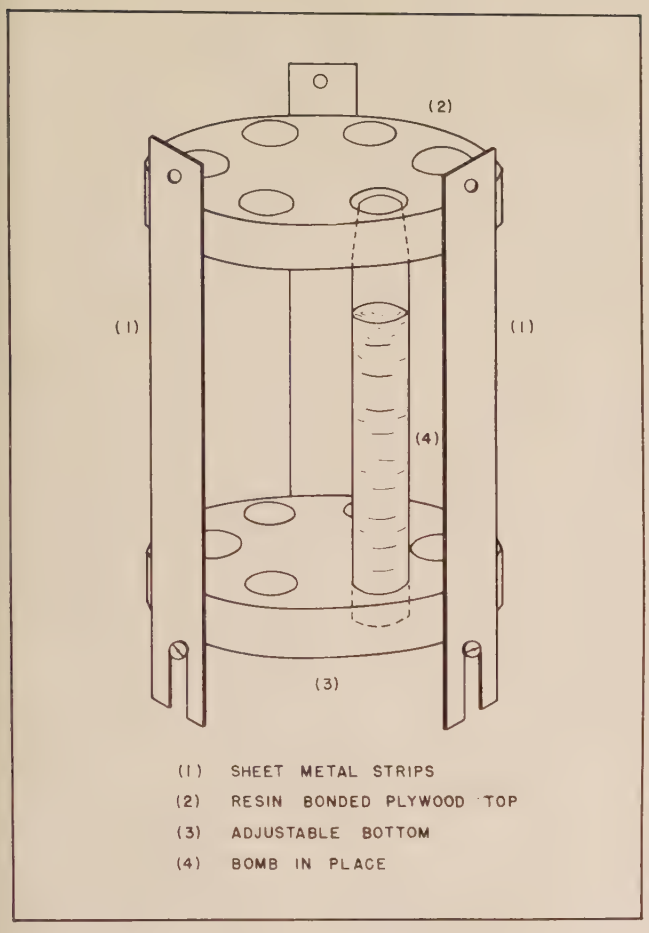

Fig. 1. Bomb rack

These trial runs showed no breakage with Pyrex glass bombs, occasional breakage with chemical-resistant glass, and somewhat higher breakage with soft glass bombs. On the basis of this performance, 1 by 8-inch culture tubes of chemical-resistant glass were used for bombs throughout the investigation. A rack for holding six glass bombs (Fig. 1) was made from 14-gage galvanized sheet-iron strips and resin-bonded plywood for top and bottom. The bottom was made adjustable to take bombs from 5 to 8 inches in length. Hangers, permanently attached to the digester cover, made it possible to fasten or unfasten the bomb rack with little delay. To open the bombs, a hot wire glass cutter served satisfactorily, although there was a tendency for bombs to explode when opened. This was caused by the carbon dioxide evolved in the reaction.

In investigations involving samples too large for the capacity of the glass bombs, another apparatus had to be used because the digester would be attacked by the acid catalysts employed. For these larger cooks, a copper autoclave (Fig. 2) was used. This autoclave was constructed to operate at pressures considerably in excess of those applied in the study. It was fitted with a steam inlet, pressure gage, sight glass, relief valve,

Table I. Results of Initial Trials With the Ploetz Determination

Sample	*Levulinic acid*	
	Ether extracted, %	*Unextracted, %*
1	26.3	17.2
2	31.0	24.4

Table II. Results of Tests on Times of Reaction

Minutes	*Levulinic acid recovered, %*	*Error, %*
5	95.32	−4.68
10	97.97	−2.03
15	97.86	−2.14
20	96.41	−3.59

and a condenser. The autoclave could also be heated by means of gas burners if necessary.

Tests of the Analytical Procedure

The determination of levulinic acid by the Ploetz and Bartels (*5*) procedure is given as follows: To a sample of 1 to 20 mg. of levulinic acid in 1 to 5 ml. of water is added 10 to 20 ml. of 0.1 *N* iodine solution. A volume of normal potassium hydroxide solution equal to that of the iodine solution is added with shaking, and the reaction is allowed to proceed for 10 minutes. At the end of this time, the iodoform is filtered off, the filtrate treated with normal hydrochloric acid equivalent to the potassium hydroxide used, and the excess iodine titrated with 0.1 *N* sodium thiosulphate.

Ploetz and Bartels investigated the influence of various factors on the results of the determination, such as temperature, time, amounts of potassium hydroxide and iodine, and interference from the presence of iodoform, formic acid, and sugars. Undecomposed sugar present in the test solution will interfere, as the sugars are subject to hypoiodate oxidation. When this condition exists, the results must be corrected for sugar on the basis of 2 iodine atoms used per molecule of glucose. Hence, a reducing sugar determination must be made. Also, when sugar is present, the amount of alkali added must be kept to a minimum (3 ml. of *N* KOH solution with 10 ml. of 0.1 *N* iodine solution).

The first attempts at using this procedure on trial cellulose hydrolysates were not entirely successful. Actual hydrolysates were filtered free from humin and divided into two parts; one half was extracted with

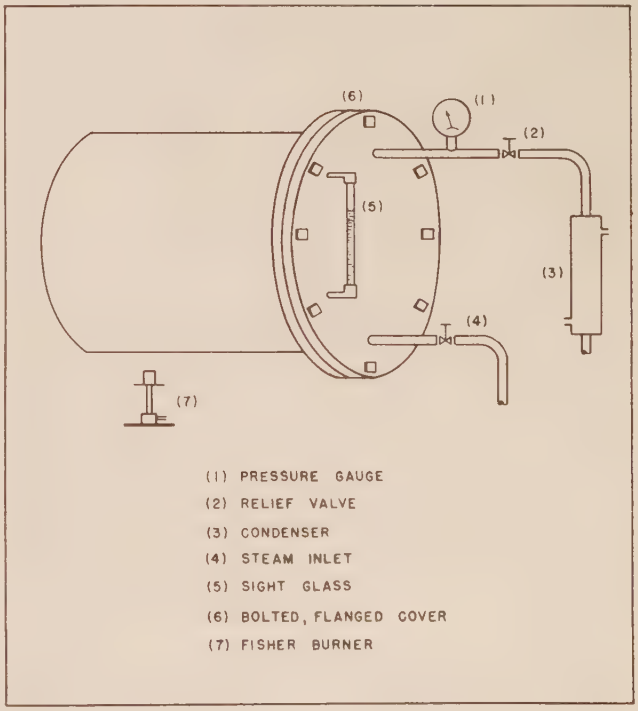

Fig. 2. Diagram of experimental copper autoclave

Table III. Data for Ploetz Determination Without Iodoform Removal

Weight levulinic acid in sample, mg.	Weight levulinic acid found, mg.	Levulinic acid, %	Error, %
9.3856	8.8191	93.97	−6.03
7.5083	7.0656	94.15	−5.85
3.6313	5.2609	93.43	−6.57
3.8195	3.5180	95.71	−6.29
1.8771	1.7815	94.91	−5.09
		Av. 94.04	−5.96

ethyl ether for 6 hours in a continuous liquid-liquid extractor. The extract was separated from the ether, made up to volume, and a Ploetz determination for levulinic acid was made. This procedure should have largely eliminated the interference from sugars present in the hydrolysate. The other half of the hydrolysate was neutralized, and determinations were made for reducing sugar by the Somogyi method (*1*) and for levulinic acid. It was assumed that when the reducing sugar correction was made, both halves of the hydrolysate should show identical percentages of levulinic acid. Actually, less levulinic acid was found in the unextracted portion of the hydrolysate than in the ether extract from the extracted portion of the hydrolysate as shown in Table I.

In order to rectify the discrepancies found, further exploratory work was performed on the analytical procedure with known samples of levulinic acid. This included tests on time of reaction, the effect of the removal of iodoform, the use of the minimum amount of potassium hydroxide, and the estimation of the equivalence value of reducing sugar. A glance at Table II will show that the literature contention as to reaction time was borne out. The actual time used in all subsequent determinations was maintained at 15 minutes. Data for the effect of the removal of iodoform are given in Tables III and IV, and the results conform to those given in the literature. The tests using the minimum amount of potassium hydroxide gave values much too low and indicated that this practice was not reliable. The data obtained on tests to find the iodine equivalence value of reducing sugar are given in Table V and indicate that large variations in iodine equivalence values may exist. Some of the differences may have resulted from the use of too highly concentrated iodine and thiosulphate solutions; i.e., 0.1 *N* solutions are too concentrated for an exact test with small amounts of reducing sugar.

The results of tests with known amounts of levulinic acid and reducing sugar present are given in Table VI. In this series of tests, glucose was added to a known levulinic acid solution to make concentrations of glucose corresponding to 40, 30, 20, and 10% of the levulinic acid present. All of the results of these tests were somewhat low, particularly the analyses using 3 ml. of potassium hydroxide, although they are computed on

Table IV. Data for Ploetz Determination With Iodoform Removal

Weight levulinic acid in sample, mg.	Weight levulinic acid found, mg.	Levulinic acid, %	Error, %
9.3856	9.1851	97.86	−2.14
7.5083	7.2460	96.51	−3.49
5.6313	5.5582	98.70	−1.30
3.8195	3.7433	99.76	−0.24
1.8771	1.9082	101.66	+1.66
		Av. 98.90	−1.10

Table V. Results of Ploetz Determinations Made on Known Glucose Solutions

Weight glucose per sample, mg.	Normal KOH and HCl, ml.	I atoms used per mol glucose
3.68	3	4.26
2.86	3	3.50
1.91	3	3.21
0.95	3	2.70
		Av. 3.42
3.68	10	3.97
2.86	10	3.74
1.91	10	4.14
0.95	10	5.62
		Av. 4.38

the basis of 6 iodines per molecule of levulinic acid. From these data, the conclusion was drawn that analyses on the unextracted hydrolysates would be too much in error to be used reliably even though their use would be advantageous insofar as economy of time was concerned. On the other hand, tests made on samples of known levulinic acid content, which were extracted for 6 hours with ethyl ether, averaged 96.25% levulinic acid recovery. This is within the error for the determination as reported in the literature.

Inasmuch as 6 hours for the ether extraction of the levulinic acid from the hydrolysates was rather lengthy and since the major portion of the acid should be extracted in less time, 4-hour extraction periods were used in subsequent work. Test solutions containing levulinic acid, glucose, and hydrochloric acid were extracted under the same conditions of time and volume in order to obtain a factor applicable to the shorter extraction time. This was found to be 0.9592. Subsequently reported percentages of levulinic acid were multiplied by the reciprocal of this factor.

Mention also should be made of the determinations of the insoluble residues in the hydrolysates. These were mixtures of unhydrolyzed cellulose and humin substance. To determine the amount of each, the residues were filtered off, oven dried at 105°C., weighed, and then subjected to additional hydrolysis with sulphuric acid in the conventional lignin determination. The material remaining on rehydrolysis was reported as humin. On the basis of the results of the foregoing tests, the analytical procedure used throughout the investigation is summarized in Fig. 3.

Influence of Factors Affecting Yield from Cellulose

The cellulose used was bleached wood pulp, HI-COLOR-G, supplied by Rayonier, Incorporated, in sheet form. It contained 89.6% alpha-cellulose and 0.064% ash. The pulp sheets were disintegrated with water in a Hobart mixer to a 10% consistency. Excess water was then removed, the pulp was allowed to air-

Table VI. Results of Ploetz Determinations of Known Levulinic Acid-Glucose Solutions

Weight levulinic acid, mg.	Weight glucose, mg.	N. KOH and HCl, ml.	Levulinic found[a], %
9.3856	3.68	10	94.6
9.3856	2.86	10	94.3
9.3856	1.91	10	95.0
9.3856	0.96	10	95.6
9.3856	3.68	3	78.6[b]
9.3856	2.86	3	77.4[b]
9.3856	1.91	3	79.3[b]
9.3856	0.96	3	79.7[b]

[a] Computed with iodine equivalent of 2 I used per mol glucose.
[b] Computed on a basis of 6 I used per mol levulinic acid.

Table VII. Percentage Yields of Levulinic Acid with Cooks of 150°C. for 1 Hour

(Values Based on Oven-dry Weight of Cellulose)

Catalyst	Levulinic acid	Formic acid	Undecomposed cellulose	Humin	Unreported by analysis
2% H_2SO_4	5.86	2.32	72.78	2.06	17.00
2% HBr	6.51	2.58	62.21	1.28	27.42
2% HCl	24.08	2.55	14.61	14.63	37.13
4% H_2SO_4	8.92	3.54	64.15	2.24	21.15
4% HBr	11.16	4.42	58.10	2.89	23.43
4% HCl	36.76	14.58	12.97	11.23	24.46
6% H_2SO_4	15.01	5.95	39.39	4.75	34.90
6% HBr	24.88	9.88	10.39	10.26	44.59
6% HCl	45.91	18.69	0.51	12.05	22.84
8% H_2SO_4	21.02	8.35	24.20	8.33	38.10
8% HBr	47.61	18.92	0.05	9.88	23.54
8% HCl	43.57	17.30	0.24	13.18	25.71

dry, and finally it was stored in a moisture-tight container.

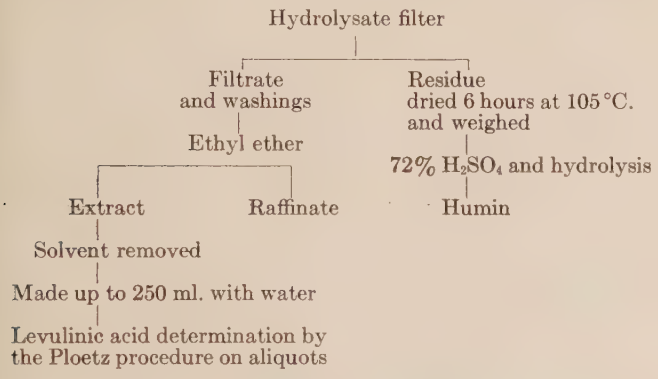

Fig. 3. Analytical procedure for levulinic acid, undecomposed cellulose, and humin

Major yield-influencing factors studied were the effects of time, temperature, catalyst, and concentration of catalyst. Lesser factors studied were the chemical to cellulose ratio and the particle size of the pulp samples. Three acid catalysts—sulphuric, hydrobromic, and hydrochloric—in concentrations of 2, 4, 6, and 8%, and certain salt catalysts were used. The temperature range selected was 150 to 190°C., with times of 1, 2, and 3 hours. It was recognized from previous work reported on wood hydrolysis that an infinite ratio of liquid to cellulose was the theoretical optimum. In this work, a 20 to 1 ratio was used throughout.

To compare the action of the three acids under the same conditions, three bombs containing the same percentage of each of the acids were heated together in the digester. Runs were made with 2% acid solutions at 150°C. for 1 hour, followed by runs using 4, 6, and 8% acid solutions kept under conditions as close as possible to 150°C. for 1 hour. This series of runs was followed by others at 170°C. for 1 hour and at 190°C. for 1 hour. When the 1-hour cooks were completed, 2 and 3 hour cooks were made, repeating the same temperature schedule. The results of the experimental cooks are given in Tables VII to XI, inclusive.

In these tables, the data in the formic acid column are calculated from the stoichiometric relationship of 1 mol. formic acid produced per mol of levulinic acid. The sum of the values listed under the headings "undecomposed cellulose" and "humin" equals the percentage total of undissolved residue. The final column, labeled "unreported by analysis," lists values obtained from the difference between the sum of the data in the other columns and 100%. It probably includes undecomposed sugars in the hydrolysate in addition to carbon dioxide and water evolved in the reaction.

Table VIII. Percentage Yields of Levulinic Acid with Cooks of 170°C. for 1 Hour

(Values Based on Oven-dry Weight of Cellulose)

Catalyst	Levulinic acid	Formic acid	Undecomposed cellulose	Humin	Unreported by analysis
2% H_2SO_4	38.31	15.19	2.91	10.33	33.26
2% HBr	44.38	17.58	1.05	9.82	27.17
2% HCl	46.85	18.52	1.34	13.62	19.67
4% H_2SO_4	40.01	15.83	2.01	12.95	29.20
4% HBr	56.25	22 27	1.18	11.98	0.32
4% HCl	41.68	16.51	1.25	13.86	26.70
6% H_2SO_4	35.79	14.10	1.78	15.43	32.81
6% HBr	50.47	19.95	0.47	11.10	18.01
6% HCl	38.00	14.28	0.36	13.76	35.60
8% H_2SO_4	29.20	11.58	1.71	16.21	41 30
8% HBr	34.25	13 59	0.01	11.87	40.28
8% HCl	30.57	12.11	1.01	19.77	36.54

Table IX. Percentage Yields of Levulinic Acid with Cooks of 190°C. for 1 Hour

(Values Based on Oven-dry Weight of Cellulose)

Catalyst	Levulinic acid	Formic acid	Undecomposed cellulose	Humin	Unreported by analysis
2% H_2SO_4	37.88	15.00	3.75	21.91	21.48
2% HBr	Bomb broken in the digester				
2% HCl	31.19	12.37	0.73	14.86	40.85

Salt Catalysts

Takahashi (*10*) has indicated that metal salts, such as zinc, copper, manganese, and ferric sulphates inhibit the formation of levulinic acid from agricultural residues. He, however, gave no quantitative data to indicate the efficiency of these salts in hindering the reactions. For this reason, experiments were carried out on cellulose using zinc chloride, arsenic trichloride, and stannous chloride.

Initially, four samples of cellulose in contact with 1, $^1/_2$, $^1/_4$, and $^1/_8$-molar-zinc chloride solutions (stabilized with hydrochloric acid) were subjected to a 1-hour cook at 150°C. Zinc chloride in a saturated solution is a cellulose solvent and, under the conditions employed, it was hoped that the foregoing solutions would partially dissolve the cellulose. This was found not to be the case, for there was little or no loss in weight of the

Table X. Percentage Yields of Levulinic Acid with Cooks of 150°C. for 2 Hours

(Values Based on Oven-dry Weight of Cellulose)

Catalyst	Levulinic acid	Formic acid	Undecomposed cellulose	Humin	Unreported by analysis
2% H_2SO_4	5.11	2.03	74.29	0.39	18.18
2% HBr	7.92	3.14	58.39	3.38	27.17
2% HCl	25.48	10.10	14.54	15.07	34.81
4% H_2SO_4	11.35	4.50	48.49	4.90	30.76
4% HBr	22.32	8.26	25 04	10.74	32.94
4% HCl	46.24	18.31	1.45	11.67	22.33
6% H_2SO_4	19.79	7.84	19.96	6.20	46.21
6% HBr	38.38	15.20	5.71	7.05	33.66
6% HCl	44.58	17.70	0.61	13.12	23.99
8% H_2SO_4	40.67	16.13	1.90	13.12	28.18
8% HBr	47.24	18 72	0 52	8.87	24.65
8% HCl	40.15	15.92	0.59	11.40	31.94

Table XI. Percentage Yields of Levulinic Acid with Cooks of 150°C. for 3 Hours

(Values Based on Oven-dry Weight of Cellulose)

Catalyst	Levulinic acid	Formic acid	Undecomposed cellulose	Humin	Unreported by analysis
2% H_2SO_4	27.50	10.90	7.19	20.88	33.53
2% HBr	36.38	14.41	4.81	8.98	35.42
2% HCl	44.62	17.71	0.90	12.32	24.45
4% H_2SO_4	35.34	13.99	9.63	11.25	20.79
4% HBr	44.16	17.51	2.46	8.51	27.36
4% HCl	42.16	16.72	0.01	11.86	29.25

cellulose after the cook. The same concentrations of zinc chloride augmented with 2% hydrochloric acid gave yields of levulinic acid considerably lower than those obtained by using 2% hydrochloric acid alone under similar conditions.

In a final experiment, 1-molar solutions of each of three salts were used in conjunction with 6% hydrochloric acid. Tin and arsenic salts hydrolyzed at this acid concentration, so the precipitates were filtered off, and the filtrate was used. The results of 1-hour cooks at 150°C. are given in Table XII. Only the tin salt had a beneficial effect, but the increase in yield would not compensate for the added cost of this catalyst.

Cooks Using Whole Wood

Inasmuch as wood sawdust should be a more economical raw material for levulinic acid production than cellulose, some preliminary runs were made on wood to get some indication of the feasibility of the process and the yields to be expected. Further, as there is pentosan material associated with wood, furfural should also be produced under the same conditions that produce levulinic acid. Thus, there is a possibility of an integrated levulinic acid-furfural process. For the investigation of these possibilities on whole-wood cooks, the copper autoclave was used.

A charge consisting of 150 grams of Douglas-fir sawdust (ground to pass through a 35-mesh screen and retained on a 60-mesh screen) and 1.5 liters of 6% hydrochloric acid was put into the autoclave. The closed autoclave was then heated by two Fisher burners until a gage pressure of 54 to 55 pounds was reached (about 10 minutes). At this point, the relief valve was opened to bleed off the furfural at the rate of 1.5 to 2.0 liters of condensate per hour. The burners were regulated to maintain the foregoing pressure. After reaching this pressure, the cook was continued for an hour. Periodically during the cook, steam was admitted to the autoclave from an external source in order to compensate for liquid losses during the bleeding-off process. At the end of the hour, the heat was removed and the autoclave pressure was slowly reduced to atmospheric pressure by means of the relief valve. Determinations for levulinic acid were made on the hydrolysate in the retort, and Tollen's phloroglucinide tests were made for furfural in the relief condensate.

Two cooks were made on whole wood; these cooks differed merely in the rates of condensate relief. In cook No. 1, reported in Table XIII, 1.5 liters of condensate were collected whereas, in cook No. 2, 2 liters were collected.

Table XII. Yields of Levulinic Acid from Cellulose Cooked 1 Hour at 150°C. in Salt-Hydrochloric Acid Solutions

(Based on Oven-dry Weight of Cellulose)

Catalyst	Yield, %
6% HCl	44.92
$ZnCl_2$ in 6% HCl	43.02
$SnCl_2 \cdot 2H_2O$ in 6% HCl	49.27
$AsCl_3$ in 6% HCl	37.70

Cook Using Cellulose in the Autoclave

Inasmuch as the yields of levulinic acid heretofore had been determined by means of the Ploetz method, a cook was made with a rather large amount of cellulose in order to isolate and to purify a sample from the hydrolysate. For this purpose, 150 grams of cellulose and 1.5 liters of 6% hydrochloric acid were placed in the autoclave and cooked for an hour at 54 to 55 pounds pressure. The details of the cook were similar to those used on whole wood except that no furfural was bled off. At the end of the cook, the residue was filtered off and washed until the washings were clear. The total solution was evaporated under vacuum to 1 liter, and this concentrate was extracted with ethyl ether for 24 hours. The ether was stripped off, leaving crude levulinic acid. This was vacuum distilled, and the fraction boiling from 135 to 143°C. at 9-mm. pressure was collected. The yield was 37.67% of the original oven-dry cellulose. The melting point, 21 to 23°C. was lower than the value of 30 to 32°C. reported in the literature. A sample of technical-grade levulinic acid, distilled in the same way, gave a product with the same melting point.

DISCUSSION

Of the three acids used, hydrochloric acid gave the highest yield of levulinic acid from cellulose in the shortest time, at the lowest temperature, and with the lowest concentration of catalyst. Under the same conditions, the maximum yield with this acid preceded the maximum for hydrobromic acid whose maximum in turn came before that of sulphuric acid. The yield with hydrochloric acid at 150°C. was approximately 45% of the oven-dry weight of cellulose (62.5% of theoretical), and the time to reach this yield was directly proportional to the percentage concentration of the acid present (Tables VII, X, and XI). At 170°C., a yield of 46.8% levulinic acid was obtained with 2% hydrochloric acid after 1 hour. (Since this work was completed, Shimizu and Takei (*8*) reported a 44.0% yield of levulinic acid from sucrose and 1.5% hydrochloric acid heated for 1 hour at 170°C.) Although the highest yield, 56.5% (78% of theoretical), was obtained with 4% hydrobromic acid and heating for 1 hour at 170°C., it is believed that this increased yield will not compensate for the higher cost of the acid. Sulphuric acid, the ideal catalyst from a cost point of view, showed inferior yields. The largest yield with this catalyst was 40.7%, obtained at 8% concentration and heating for 2 hours at 150°C. This yield was about the same as that from

Table XIII. Yields of Levulinic Acid and Furfural from Whole Wood Cooked 1 Hour at 150°C.

Cook number	Levulinic acid, based on oven-dry weight of wood, %	Levulinic acid, based on 55% cellulose in wood, %	Furfural, based on oven-dry weight of wood, %
1	23.80	43.27	0.85
2	20.58	37.42	1.59

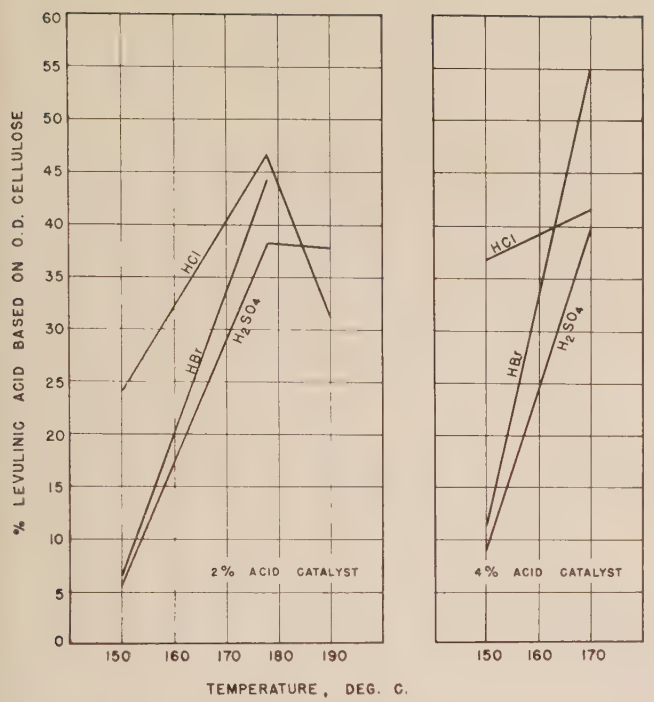

Fig 4. Relationship between temperature, catalyst concentration, and levulinic acid yield at a cooking time of 1 hour

cellulose and 4% sulphuric acid heated for 1 hour at 170°C.

The experimental data seem to reveal an interesting cyclic tendency. At a 2% acid catalyst concentration, all catalysts showed a sharp increase in yield of levulinic acid as temperatures were increased from 150 to 170°C. However, at 4% the hydrochloric acid showed a less sharp increase in going to 170°C., whereas the hydrobromic and sulphuric acids still showed high rates of increase (Fig. 4).

At a 6% acid catalyst concentration, the yield with hydrochloric acid actually showed a decrease from 150 to 170°C., and the rate of increased yield from hydrobromic acid declined. The yield with sulphuric acid was still increasing at a good rate (Fig. 5).

With 8% catalysts and increasing temperature, both hydrochloric and hydrobromic acids showed decreased yields, and the rate of increased yield with sulphuric acid diminished. With 2% catalysts again, both the sulphuric acid and hydrochloric acid showed decreased yields in going from 170 to 190°C., and it is assumed that the yield with hydrobromic acid also would have showed a decrease had the bomb not broken. Thus, the cycle seems to be hydrochloric acid reaching the optimum yield point first, followed by hydrobromic acid and then by sulphuric acid. From the data, it appeared that temperatures above 190°C. and concentrations of more than 2% at 190°C. were not worth while investigating.

The similarity in yields of levulinic acid at 150°C. with a 2% catalyst at 1 hour (Table VII) and 2 hours (Table X) is not explained. A rerun of the 2-hour cook was made, but the values of the two runs did not differ by more than 1%.

The results in Tables VII, X, and XI show the yields of levulinic acid as functions of time and concentrations of catalyst. The yields with hydrochloric acid were rather uniform for different combinations of catalyst concentration and time, indicating that an operator of the process easily could alter the condition to suit the economic factors prevalent at the time. For example, should a lower concentration of catalyst be advisable from the viewpoint of all-around economy, a longer time with a dilute catalyst would be used. On the other hand, at a higher concentration of catalyst, a shorter reaction time would be used. Hydrobromic acid and sulphuric acid gave less consistent results.

The data in Tables VII through XI on the percentage of undecomposed cellulose remaining in the cooks substantiate most of the foregoing conclusions. There is a direct relationship observed between yield of levulinic acid found and percentage of undecomposed cellulose remaining. In cooks at low temperatures and low catalyst concentrations for short times, the amount of undecomposed cellulose was relatively large. With harsher cooking conditions and longer times, this cellulose tends to be completely hydrolyzed, and the amount of humin becomes greater. Again, a cycle is apparent wherein complete cellulose hydrolysis occurred first with hydrochloric acid cooks and last with sulphuric acid cooks. Humin formation increased with increase in yield of levulinic acid. For similar levulinic acid yields, sulphuric acid produced the greatest amount of humin and hydrobromic acid the least amount. In general, the better over-all yield of levulinic acid by the use of hydrobromic acid may be attributed to the fact that this acid has probably the least degradative action during the reaction.

The final column in the tables, labeled "unreported by analysis," shows high values in cooks using mild conditions, low values at optimum levulinic acid yields, and high values again in cooks using the most harsh conditions. The high values in the mild cooks can be

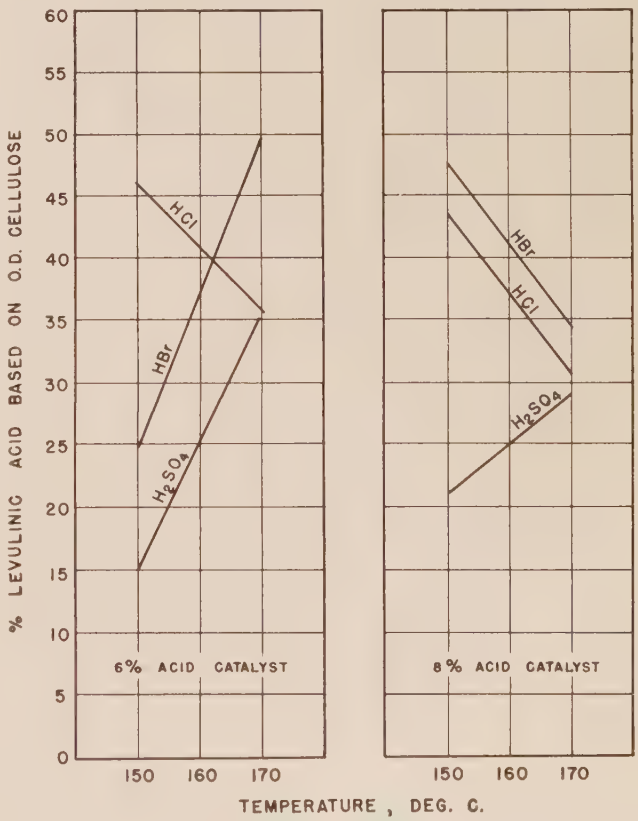

Fig. 5. Relationship between temperature, catalyst concentration, and levulinic acid yield at a cooking time of 1 hour

attributed to sugar present in the hydrolysate, whereas the high values under the harsh conditions are presumably caused by degradation products. In these cooks, the reaction bombs tended to explode with so much force upon opening that a special bomb opener had to be used in order to avoid loss of the sample.

The cooks on whole wood showed that this material could serve as a starting material for levulinic acid manufacture. Also, it appears that an integrated process for the manufacture of levulinic acid, formic acid, and furfural is feasible. For this purpose, hardwoods should be more suitable than softwoods which are usually lower in cellulose and furfural-yielding pentosans.

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Correlation of B.O.D. and Chemical Tests on Industrial Effluents

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In a study on the combined effluents from a kraft pulp and paper mill and a hardwood distillation plant, statistical techniques have been used to determine the correlation between 5-day B.O.D. values and data from tests on oxygen consumed by permanganate and total solids.

Numerous attempts have been made to develop methods for testing the oxygen demand or oxygen consuming power of human and industrial wastes. The biochemical oxygen demand (*1*), or so called 5-day B.O.D. test, has been accepted as the standard method by most investigators. It is defined as the oxygen in parts per million required during stabilization of the decomposable organic matter by aerobic bacterial action under standard specified conditions. The results of the 5-day B.O.D. test seem to give more information concerning the strength of wastes and their oxygen depleting potentials in natural streams than any other single laboratory test. The most serious objection to this test is the length of time required. For plant control in many industrial waste systems, such values are of use only as a matter of record. By the time B.O.D. values have been obtained it is usually too late for any preventive action. For this reason some quicker method of determining oxygen consuming potentials of industrial wastes is a necessity.

The oxygen consumed from permanganate test, given in the "Standard Methods for Examination of Water and Sewage" (*2*), is an example of a chemical test that has been used for estimating the strength of industrial wastes. Other chemical oxidizing agents that have been proposed are potassium dichromate, ceric sulphate, and iodic acid. A discussion of the various methods is given in a recent article by Moore, et al. (*3*). It is known that chemical methods for estimating the oxygen-consuming potentials of wastes do not give values identical to B.O.D. values because chemical oxidation methods do not differentiate between biologically stable and unstable forms of organic matter. Nevertheless some method of quickly estimating the strength of industrial wastes is a necessity and as a result many different chemical tests are in use. An investigator facing the task of selecting the most suitable of these chemical tests for an industrial waste is presented with a most serious problem. To date, adequate data are not available in the literature to properly evaluate the various proposed methods on specific industrial wastes. The results of one study made in the Research Laboratory of the Crossett Lumber Co. is reported herein. It is believed that similar studies made in other laboratories should be published to aid in evaluating the various methods.

The industrial wastes of concern in this study were those from a kraft pulp and paper mill and a hardwood distillation plant. The combined waste effluent amounted to approximately 12,000,000 gallons daily. The wastes were those incident to the manufacture of natural and bleached kraft pulp and paper, charcoal, acetic acid, and methanol. All by-products such as tall oil, sulphate turpentine, and wood creosote oils were recovered for sale in as efficient a manner as practical. Of the total B.O.D. in pounds per day the pulp and paper mill contributed approximately 85%.

PROCEDURE

In this study, daily composite samples were taken during the period of July 7 to Nov. 15, 1949. A study of these wastes indicated that there were no toxic materials which were deleterious to aquatic life or that appeared to interfere with the B.O.D. tests. During this period,

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54 samples were analyzed in the laboratory completely for B.O.D., oxygen consumed from permanganate, total solids, and volatile solids. These samples were taken over a sufficiently long enough period of time to include all normal and several abnormal periods of plant operation. Therefore, it is believed that the range covered is sufficient to justify a thorough study of data.

Composite samples were obtained by a continuous sampler in the effluent ditch just below the point where all the plant sewers combined. The samples were collected each morning and immediately brought into the laboratory for analysis. The procedures used for all tests were those given in the "Standard Methods for Examination of Water and Sewage," ninth edition. The modifications used in the B.O.D. test were those recommended by the National Council for Stream Improvement (*4*). In the oxygen consumed from permanganate test 0.0125 *N* permanganate was used for digestion.

Table I

B.O.D. *y*	*Oxygen consumed from permanganate* *x*	*Total solids* *t*
87	76	945
99	73	752
82	146	1037
96	162	1039
172	368	1308
130	250	1190
189	290	1218
95	128	981
134	171	1070
186	175	1239
186	240	1349
127	178	1282
223	216	1306
277	338	1418
265	323	1355
215	420	1484
223	330	1060
247	280	1283
216	243	1181
192	213	1134
145	175	986
105	120	893
134	155	988
156	175	1170
136	190	1114
117	143	1023
103	120	966
148	185	1167
298	345	1667
199	270	1331
191	278	1240
183	225	1170
214	255	1181
316	380	1476
302	368	1689
318	358	1527
247	600	1857
180	440	1474
192	295	1032
267	370	1211
252	395	1406
318	590	1676
295	460	1478
266	505	1530
291	440	1603
332	490	1492
346	490	1575
381	570	1605
260	405	1315
156	285	1083
374	550	1622
244	365	1378
269	450	1461
413	615	1725
Av. 214.61	308.83	1291.52

The statistical techniques of correlation and regression were used to determine the degree of correlation between the B.O.D. values and the tests of oxygen consumed, total solids, and volatile solids. These statistical techniques are described in standard texts on the subject. For those not familiar with statistical methods it can be said that they are used to determine mathematically the following:

1. The best straight-line relationship between two variables. This is called a regression line.
2. The correlation coefficient. This number indicates the degree of correlation between two variables.
3. The variance about the regression line or the measure of error variance of any single observation. This value allows us to estimate the possible error of our predicted B.O.D., we record an interval, say 200 ± 80, and a probability, say 0.95. This means that the probability of the true B.O.D. value lying between 120 and 280 is 0.95. Thus, 200 ± 80 is called a 95% confidence interval.

It was found that a highly significant correlation did exist between the B.O.D. values and the oxygen consumed values, also between B.O.D. and total solids. The correlation between B.O.D. and volatile solids was not as good as the others. Using the technique of multiple correlation it was found that the B.O.D. could be correlated to both oxygen consumed and total solids better than to either alone.

RESULTS

The data obtained on the analysis of 54 daily composite samples for B.O.D., oxygen consumed (O.C.), and total solids (T.S.) are given in Table I.

A summary of these data presented in Table II shows that samples ranging in B.O.D. from 82 to 413 were included in the study. The limits computed from the standard deviation show that 99.7% of our values will lie within 34.1 and 463.3 B.O.D. with the plants operating as they were during this test period. This mean and range furnishes a sound statistical basis to evaluate future plant changes and to tell when either the mean B.O.D. of the effluent is lowered or the operating range is decreased. If desired, a quality control chart can be set up to determine when the "quality" of the effluent is improved and to detect when the system is "out of control."

For the purpose of determining degree of correlation between B.O.D., oxygen consumed and total solids the following calculations were made:

Let y = B.O.D. in parts per million
x = oxygen consumed from permanganate in parts per million
t = total solids in parts per million

The regression line of y on x is determined by the method of least squares. That is, if $y = ax + b$ is the equation for the regression line, a and b are found by solving the two simultaneous equations

$$(\Sigma x)a + nb = \Sigma y$$
$$(\Sigma x^2)a + (\Sigma x)b = \Sigma xy.$$

Table II

Value	*Number of samples*	*Mean*	*Standard deviation*	*Range* *High*	*Range* *Low*
B.O.D.	54	214.61	82.9	413	82
O.C.	54	308.83	141.8	615	73
T.S.	54	1291.52	244.5	1856	752

The correlation coefficient is

$$r = \frac{\Sigma xy - \frac{(\Sigma x)(\Sigma y)}{n}}{\sqrt{\left[\Sigma x^2 - \frac{(\Sigma x)^2}{n}\right]\left[\Sigma y^2 - \frac{(\Sigma y)^2}{n}\right]}}$$

The variance about the regression or the measure of the error variance of any single observation is determined by the equation

$$S_R^2 = (1 - r^2)\left[\frac{\Sigma y^2 - \frac{(\Sigma y)^2}{n}}{n - 2}\right]$$

The standard deviation of predictions of B.O.D. from O.C. $= S_R$. The 95% confidence interval for $y = ax + b \pm 2S_R$.

Data on the correlation of x, y, and t are given in Table III. Reference to tables (*5*) shows that the correlation coefficients in both cases are highly significant. This means that within the B.O.D. range studied there is a high degree of correlation between B.O.D., oxygen consumed, and total solids values. B.O.D. can be calculated from oxygen-consumed data with assurance that it will be within 80 p.p.m. of the true value 95% of the time. B.O.D. can also be calculated from total solids with an accuracy of ±90 p.p.m.

Since a high degree of correlation is obtained between y and x and also between y and t, it is to be expected that x and t could be combined to give an even better estimate of y. By extending the calculation to a regression plane instead of a line (*6*), we find that B.O.D. can be calculated from oxygen consumed and total solids by means of the equation

$$y = 0.349x + 0.107t - 32.$$

The correlation coefficient in this case is 0.893, the standard deviation is 39 p.p.m., and the 95% confidence interval is the calculated y value ± 78 p.p.m.

It is apparent that the absolute values of B.O.D. calculated from oxygen consumed and total solids tests will need to be used with caution and discretion. With the limits of error known, however, they can be used with confidence. In many cases only the upper limits of B.O.D. are of interest since we may be concerned in knowing when a danger zone is approached. If the limits of error of the tests are known, appropriate measures to check laboratory results can be taken at the proper time.

It should be pointed out that the reproducibility of the B.O.D. test itself is not good. It is well known that the limits of error attached to a single B.O.D. determination are appreciable. Ingols, Hildebrandt, and Ridenour (*7*) have made an extensive study of the accuracy and reproducibility of the B.O.D. and other tests. Their results show that the variations obtained in the B.O.D. test exceed by a considerable amount the 10% variation which is generally accepted as the accuracy. In one series of tests on blended raw sewage with an average B.O.D. of 225 p.p.m., they have an average probable error of each individual value of ±22. This would correspond to a standard deviation of 32.6 p.p.m., and should be compared to the standard deviation of 39 p.p.m. found in this work. Others have found larger and smaller deviations. There is no doubt that the limits of error in the B.O.D. test will vary depending on the sewage or waste studied and the techniques used. They would need to be determined in each individual case. Unfortunately, data are not available to establish these limits on the waste in this study. It is clear, however, that the error involved in B.O.D. determinations is appreciable and should be taken into consideration in evaluating this and any other method of calculating B.O.D. from chemical tests. While the standard deviation or the confidence interval shown in this work is quite large, it is probably not appreciably greater than would be found in determining true B.O.D. from any single B.O.D. determination.

Table III

Equation for calculated B.O.D. from oxygen consumed	$y = 0.516x + 55$
Equation for calculated B.O.D. from total solids	$y = 0.287t - 156$
Correlation coefficient for B.O.D. from oxygen consumed	$r_{xy} = 0.882$
Correlation coefficient for B.O.D. from total solids	$r_{yt} = 0.848$
Standard deviation for B.O.D. from oxygen consumed	$S_R = 40$
Standard deviation for B.O.D. from total solids	$S_R = 45$
95% confidence interval for B.O.D. from oxygen consumed	$y \pm 2S_R = 0.516\,x + 55 \pm 80$
95% confidence interval for B.O.D. from total solids	$y \pm 2S_R = 0.287\,t - 156 \pm 90$

CONCLUSIONS

From this study it can be concluded that within the range of 34 to 463 p.p.m. the B.O.D. of the industrial waste studied can be calculated from the values of oxygen consumed from permanganate and total solids.

The calculated values of B.O.D. will, 95 times out of 100, fall within the following limits:

Using oxygen-consumed values ± 80 p.p.m.
Using total-solids values ± 90 p.p.m.
Using both values ± 78 p.p.m.

With the limits of accuracy thus known, it is possible to calculate B.O.D. from quicker laboratory tests and to set up a control which can be used with confidence.

It is believed that a statistical analysis of stream pollution testing data will enable investigators to better evaluate proposed improvements in laboratory methods and plant operations.

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Crystalline Hemicelluloses

I. Crystalline and Amorphous Xylan from Barley Straw

ALBERT P. YUNDT

COLD Fehling solution selectively precipitates the xylan-containing chains of hemicelluloses (*1*). For straw, this fraction has a degree of polymerization of 150 (*2*), and is composed (*3*) of D-xylose, L-arabinose, and a uronic acid in the molar ratio of 23:0.9:1. Whenever investigated, combined L-arabinose has always been found in nature in the furanose ring form, whereas D-xylose occurs in the pyranose form (*4*). Furanosides are much more readily hydrolyzed by acids than pyranosides (*5*). By a mild acid hydrolysis of esparto hemicellulose (which contains only xylose and arabinose), Haworth (*6*) was able to remove the arabinose completely and leave the xylan chain portions unaffected. It seemed likely that straw hemicelluloses might behave similarly, possibly even yielding fragments free of uronic acids. As pilot experiments showed promise, the experiment was tried on a large scale so that the products could be analyzed.

In the work to follow, analyses, where applicable, are by standard methods. Pentosan and uronic acid analyses, however, have been corrected for the effect of the one material on the determination of the other (*7*, *8*).

PREPARATION OF THE XYLAN-RICH HEMICELLULOSE FRACTION

Mature barley straw was reduced to 36 to 60 mesh and was chlorited in the usual fashion to a deliberately high lignin content of 4.5%. The hemicelluloses were extracted with boiling 5% sodium hydroxide solution. They were recovered and purified by the Fehling solution method of Salkowski (*1*) which was repeated twice. The last traces of copper were removed by electrodialysis against tap water. Analyses of this and the intermediate materials are given in Table I.

Table I. Analyses of Products in the Preparation of Barley Hemicelluloses

	Barley straw	*Barley holocellulose*	*Extracted residue*	*Electrodialyzed hemicelluloses*
Lignin (ash free), %	21.35	4.50	...	...
Ash, %	1.49	1.32	1.39	1.36
Uronic anhydride (cor. for cellulose and xylan), %	3.45	2.44	0.34	6.47
Pentosans as xylan (cor. for cellulose and uronic anhydride), %	25.4	28.6	8.4	89.5
Cellulose (by difference), %	48	63	90	...

Selective Acid Hydrolysis of the Hemicelluloses

The hemicellulose solution, heated in a boiling water bath, was made 0.2% in oxalic acid. The reducing value of the solution rose rapidly at first but became virtually constant at the end of 5 hours, showing that all the more susceptible linkages had been hydrolyzed. The total time of hydrolysis was 6.5 hours. A highly hydrous, water-insoluble fraction (fraction A) could be isolated only after partial dehydration by the addition of one volume of ethanol. Fraction B was recovered by concentrating the liquors and precipitating the sirup into acetone. The ash of fraction A was largely calcium oxalate, the result of using tap water in the electrodialysis. In a second preparation, this material was mechanically separated after centrifuging. The analyses of the two fractions are given in Table II and yields at each step in the preparation are given in Table III.

Table II. Analyses of Fractions A and B

	Fraction A		*Fraction B*
	Preparation 1	*Preparation 2*	*Preparation 1*
Ash, %	5.98[a]	0.85	1.54
Uronic anhydride (cor. for CO_2 from xylose), %	2.20	2.41	7.55
Methoxyl, %	0.39	0.43	...
Pentosans as xylan (cor. for uronic anhydride), %	92.6	97.4	84.1
Pentosans as xylan (ash free), %	98.5	98.2	85.4
Methyl uronic anhydride (ash free), %	2.52	2.62	8.3
Total, %	101.0	100.8	93.7

[a] CaO and MgO content calculated to calcium oxalate.

Properties of Fractions A and B

Fraction B was highly water soluble. The degree of polymerization by the Munson-Walker reducing value is about 8, whereas analysis indicates a ratio of about 1 uronic acid to 15 xylose units. It either contains two reducing groups per molecule or is a mixture. The

Table III. Yield of Products

Material and source	*Yield for operation*	*Yield based on straw*
Holocellulose from straw, %	87.7	87.7
Purified hemicellulose from holocellulose, %	22.3	19.5
Extracted residue from holocellulose, %	71.1	62.3
Losses in extraction and purification, %	6.6	5.8
Fraction A from hydrolysis of hemicellulose, %	37.8	7.4
Fraction B from hydrolysis of hemicellulose, %	43.3	8.5
Not recovered from hydrolysis of hemicellulose, %	18.9	3.7

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latter seems the more likely for the uronic acid analyses indicated inhomogeneity of the sample. In water solution, $[\alpha]_D^{20} = -50.8°$. It was not investigated further.

Fraction A was insoluble in water but, when freshly prepared, would dissolve in 3 *N* sodium hydroxide. On aging or momentary contact with very dilute alkali,

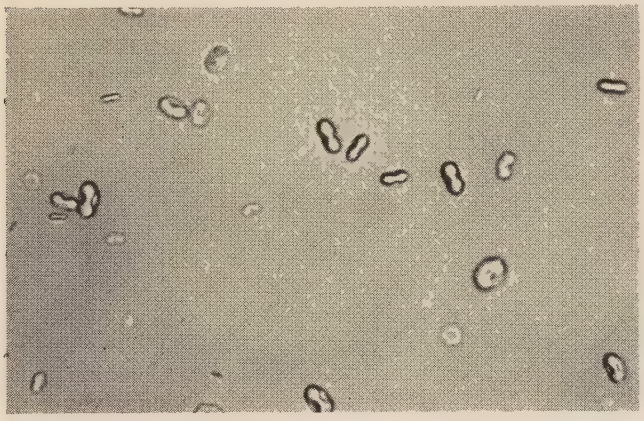

Fig. 1. Barley straw xylan mounted in glycerol-water 750 ×

it lost its alkali solubility and the ability to swell in water. Solution in concentrated phosphoric acid and reprecipitation restored these properties, but the phosphoric acid was difficult to remove completely.

PREPARATION OF CRYSTALLINE XYLAN

Complete sodium hydroxide solubility, but not the water swelling property, is regained on autoclaving a water paste of fraction A at 120°C. for 15 minutes. Prolonged (3 to 5 hours) autoclaving of such a paste at pH 5 to 6 results in the formation of spherocrystals of xylan large enough to filter, and most of the uronic acid-containing material remains in solution in the surrounding liquor. The xylan was restored to the amorphous state by dissolving in alkali, filtering, and

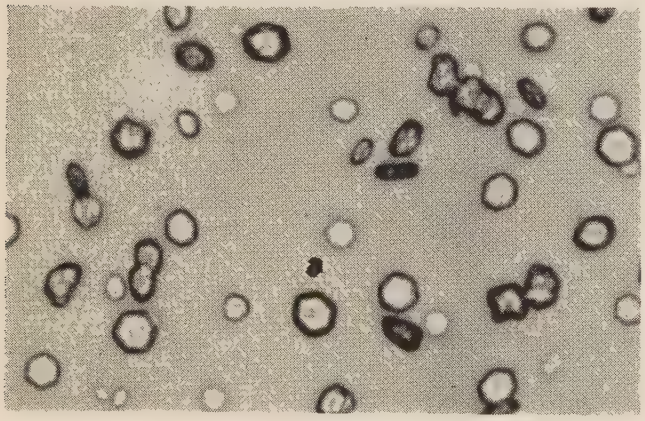

Fig. 2. Barley straw xylan mounted in Nephrax 750 ×

precipitating with acetic acid and was then reautoclaved. Three such crystallizations sufficed to reduce the uronic anhydride content of one sample to 0.4%. The ash content was 0.34% and pentosans content (calculated as xylan) was 99.5%.

The purified amorphous material forms a clear water solution with a concentration of 0.2% at 120°C. On cooling to 60 to 70°C., the xylan crystallizes as hexagonal platelets with rounded corners as shown in Figs. 1 and 2. Some of the crystals in Fig. 2 are on edge and have been "optically cross sectioned" by the limited depth of the field of the microscope. It will be noted that they are dumbbell shaped in cross section. Figure 3 is an electron micrograph of the crystals; this shows somewhat more clearly the somewhat rounded corners of the crystals. This is believed to be the first report of a crystalline xylan.

Physical Properties of the Crystals

The crystalline xylan will absorb 10 to 15% of moisture. Dry crystals show typical crystalline x-ray diffraction patterns. Moist crystals show less sharp maxima and the characteristic spacings are increased about 7.5%, showing that the water molecules penetrate the structure and distort the crystal lattice.

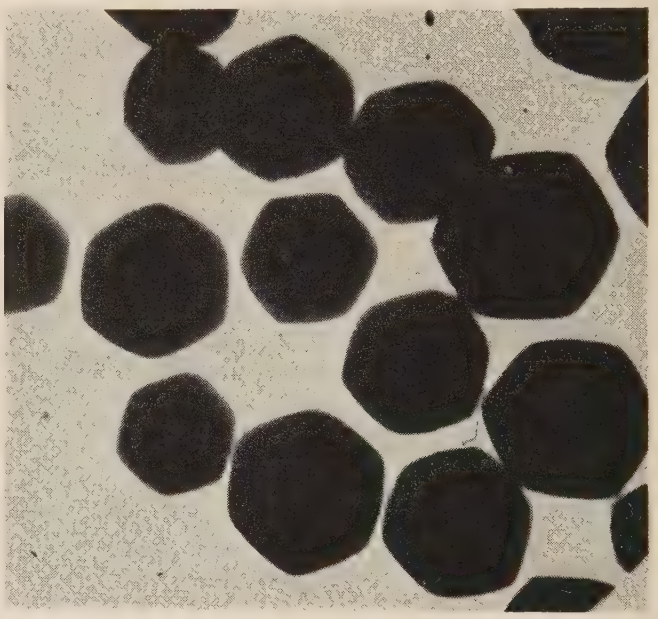

Fig. 3. Electron micrograph of barley straw xylan crystals

Crystals seen on edge between crossed Nicols show double refraction; alternate maxima and extinctions are visible every 45° of rotation. When viewed perpendicular to the plane of the platelet, optical activity was noted only in impure crystals. These show typical strain patterns. The spherocrystals also showed double refraction effects.

Proof of the Xylan Nature of Crystals

Crystalline D-xylose was prepared in good yield, and its identity confirmed. A diphenylhydrazine test for arabinose in the mother liquor was negative. This, coupled with the high pentosan analysis previously mentioned, indicates relatively pure xylan. Xylose determinations on 3% nitric acid hydrolyzates (to reduce furfural formation) by the method of Breddy and Jones (*9*) as modified by Wise and Ratliff (*10*) and that of Wise and Appling (*11*) confirmed this finding. Since this work was completed, Chandra *et al.* have reported the preparation of a xylan, from esparto grass, free from residues other than xylose (*12*).

Derivatives

Xylan dinitrate and diacetate were prepared and found to be insoluble in all neutral solvents tested. Methylxylan was prepared in a nitrogen atmosphere. It was insoluble in ether and soluble in chloroform, acetone, and cold, but not hot, water.

Molecular Weight

The osmotic pressure of chloroform solutions of methylxylan was determined at concentrations of 4, 2.5, and 0.5 gram per liter. The indicated molecular weights, when extrapolated to zero concentration, corresponded to a degree of polymerization of 39.2. Aldose end-group determinations were made on amorphous and crystalline xylan samples by a new method. Assuming one aldose end group per molecule, the average of the two determinations indicates the same degree of polymerization.

Optical Rotations and Structure

Table IV gives the optical rotations for the xylan and methylxylan, together with values from the literature for Haworth's esparto xylan (*6*).

Table IV. Optical Rotations

Substance	*Concentration, g./l.*	*Solvent*	$[\alpha]_D$	$[\alpha]_{436}$
Haworth's esparto xylan	..	6% NaOH	−111°	...
Barley straw xylan	43.2	6% NaOH	−106°	−204°
Haworth's methylxylan	..	Chloroform	−90 to −92°	...
Barley methylxylan	24.5	Chloroform	−91°	...
Barley straw xylan	4.9	TAPPI cupram-monium	...	−1635°

Since the barley xylan values agree quite well with those for Haworth's esparto xylan, which he has shown to be predominantly 1,4′-β-linked xylopyranose units, barley xylan most probably has predominantly the same linkage. The rotational shift method of Reeves (*13*) gives strong support to the existence of this linkage in barley xylan. His molar shift for the xylan is calculated as −188, 900°, as compared with −191, 200° for cellulose.

Simplified Method of Preparation of Crystalline Xylan

Purification of the hemicellulose prior to selective hydrolysis was found to be unnecessary. A crude dark brown barley hemicellulose preparation was made into a water paste at pH 4.0 and autoclaved 4 hours at 120°C. This simultaneously accomplished the selective acid hydrolysis and the first crystallization. Most of the colored impurities polymerized and could be filtered after dissolving the spherocrystals in alkali. Two more crystallizations sufficed to give a relatively pure xylan.

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Crystalline Hemicelluloses

II. Crystalline Xylan from Paper Birch

ALBERT P. YUNDT

The purified hemicelluloses of European beech have been shown to possess essentially a uniform degree of polymerization of 150 (*1*) and a molar ratio of sugar units of approximately 2 L-arabinose to 16 D-xylose to 1.1 uronic acid (*2*, *3*). Comparable data are not available for American woods, but the hemicelluloses of paper birch are probably quite similar. Accordingly, it seemed likely that a crystalline xylan could be prepared from paper birch using the techniques that were employed for barley xylan.

PREPARATION AND SELECTIVE ACID HYDROLYSIS OF THE HEMICELLULOSES

The xylan-rich hemicellulose fraction of paper birch was prepared by methods analogous to those used for barley straw described in part I. The analyses of the various materials are given in Table I. The selective acid hydrolysis was also similarly conducted with similar results. The reducing value per gram of hemicelluloses was about 30% higher than that for straw at the end of the hydrolysis and a higher ethanol concentration (65%) was required to facilitate handling the precipitate. The dried precipitate (fraction A) was quite impure as indicated by a high uronic anhydride content (6.4%). It was not analyzed further, but was subjected to purification by crystallization. Recovery of a fraction B from the hydrolyzate was not attempted. The yield data are given in Table II.

CRYSTALLIZATION OF BIRCH XYLAN

The purification of birch xylan by crystallization

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proved more difficult than that of barley xylan. In general, the birch xylan seemed to have properties more nearly similar to those of the impurities and was

Table I. Analyses of Products in the Preparation of Birch Xylan

	Wood meal	*Birch holocellulose*	*Electrodialyzed hemicellulose*	*Xylan crystals*
Lignin (ash free), %	20.7	3.01	...	...
Ash, %	0.24	0.54	1.19	0.40
Uronic acids (cor. for cellulose and xylan), %	...	2.77	8.2	2.40
Pentosans as xylan (cor. for cellulose and uronic anhydride), %	28.0	33.8	84.6	98.4
Cellulose (by difference), %	50	60	...	...

more difficult to crystallize. A cyclic purification scheme was used, wherein the material which did not crystallize after each successive autoclaving was recovered by addition of 1.5 volumes of ethanol and was treated as if it were starting material for the purification. After three spherocrystal purifications by 4-hour autoclaving, the material was recrystallized from a 0.2% water solution formed at 120°C. The analysis of the material is included in Table I. Crystallization yields were not included in Table II because of manipulative losses. A 3% nitric acid hydrolyzate was bioassayed by the method of Wise and Appling (*4*). The unfermentable uronic acids gave an unweighable trace of cuprous oxide and, within the limits of experimental error, all sugars present were fermented. In consideration of the high pentosan content, it would seem that xylose and uronic acids are the only two constituents.

Viscometric comparison with barley xylan indicated a degree of polymerization of 35 for birch xylan. Optical rotational measurements gave results as follows: for a 33-gram per liter solution in 6% sodium hydroxide, $[\alpha]_D^{30} = -92°$ and $[\alpha]_{436}^{30} = -196°$; for a 5-gram per liter solution in TAPPI cuprammonium solution, $[\alpha]_{436}^{30} = -1630°$. Reeves' molar rotational shift (*5*) is calculated at $-189{,}000°$ as compared with $-181{,}200°$ for cellulose, which again indicates essentially 1,4′-β-linked xylopyranose units.

Table II. Yield of Products

Material and source, %	*Yield for operation*	*Yield based on wood*
Holocellulose from wood meal	74.5	74.5
Electrodialyzed hemicellulose from holocellulose	24.9	18.6
Fraction A from hydrolysis of hemicellulose	39.8	7.4

The crystal habit and characteristics are the same as for barley xylan. The two preparations seem to differ from each other mainly in the purity of the product.

Attempts to prepare a slash pine xylan by similar procedures were unsuccessful. After selective hydrolysis, all fragments were soluble in 65% ethanol.

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Crystalline Hemicelluloses

III. Acid and Enzymatic Hydrolysis of Xylans

ALBERT P. YUNDT

Decay of wood and straw usually involves hydrolysis of the hemicelluloses by extracellular enzymes. The resultant simple sugars generally furnish a large part of the nutritional requirement of the organism causing the decay. The general question was discussed previously in connection with cellulose (*1*). Previously reported work on the enzymatic hydrolysis of xylan has been on impure material containing a high percentage of uronic acids and probably some arabinose, with the result that the data are difficult to interpret. The sources of hemicelluloses and hemicellulases used varied widely, but the optimum pH found for hydrolysis was from 4.5 to 5.0—usually 4.65. Since a pure xylan was now available from straw or birch (see preceding paper of this series), it seemed desirable to investigate the hydrolysis of the pure substrate.

The same *Aspergillus niger* mixed enzyme source, precautions, and methods of preparation were used as were described for the enzymatic hydrolysis of cellulose (*1*). The extent of hydrolysis was estimated by determining the cyanide consumption of the aldose end groups.

The optimum pH for the enzymatic hydrolysis was found to be 5.8 or higher. The exact optimum is questionable because, at higher pH's, the enzyme system begins to precipitate. This information, however, was not obtained until completion of the other work. The other experiments were conducted at pH 4.65, the optimum found by previous workers with their impure mixed substrates. A change of pH would be expected to alter only the speed of the reaction.

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ENZYMATIC HYDROLYSIS OF AMORPHOUS XYLAN

A sample of freshly precipitated, highly purified barley straw xylan was made into a stock suspension and samples were enzymatically hydrolyzed at pH 4.65 and 45°C. for varying times. The results shown in Fig. 1 demonstrate that an extremely rapid initial

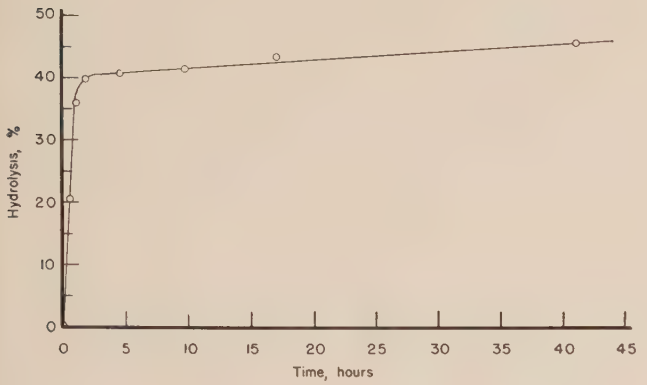

Fig. 1. Enzymatic hydrolysis of barley xylan

hydrolysis occurs, followed by a very slow reaction on the resistant remainder. The residue may be said to have retrograded by analogy to a similar phenomenon observed in the enzymatic hydrolysis of amylose. It can be reactivated by wet autoclaving at 120°C.

ENZYMATIC HYDROLYSIS OF CRYSTALLINE XYLAN

Samples of xylan crystals were treated in the same way as the amorphous material with much the same

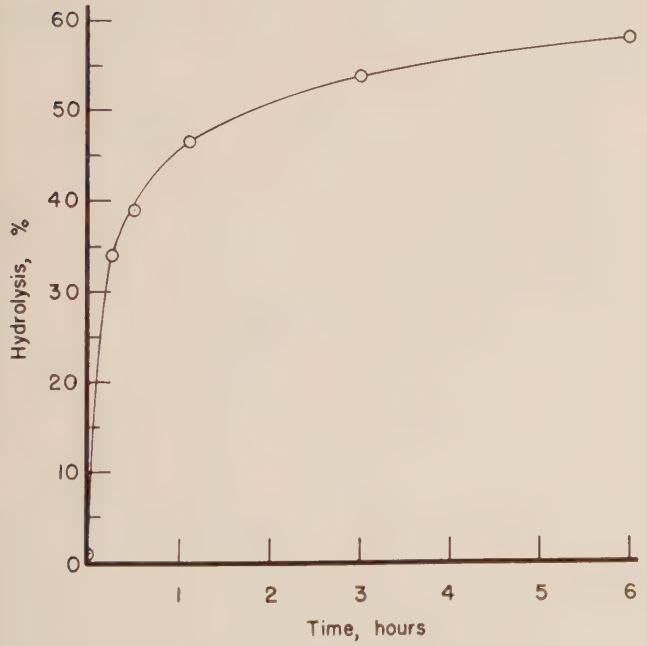

Fig. 2. Enzymatic hydrolysis of barley straw hemicellulose

results. The curve is similar in shape to that for the amorphous xylan. The initial hydrolysis, however, was much faster—so fast that the methods used did not suffice to accurately measure it—and the subsequent slow hydrolysis was much slower. A sample showed essentially the same degree of hydrolysis in 1 hour as in 24. Samples varied greatly in the limiting degree of hydrolysis, from less than 10% to more than 90%. In general, the less highly purified samples seemed to give higher degrees of hydrolysis, but attempts to prove this point by deliberate readdition of recovered impurities prior to crystallization gave negative results. The same crystalline sample that showed 90% hydrolysis also showed, after solution and precipitation, the highest hydrolysis of amorphous samples (78%). These same crystals, when dried and subsequently soaked in water for 24 hours, gave a limiting hydrolysis of 50%. Contact with dilute alkali will also retrograde the material.

To show that the retrogradation was a physical influence on the enzymatic reaction rather than representative of chemical inhomogeneity, a sample was

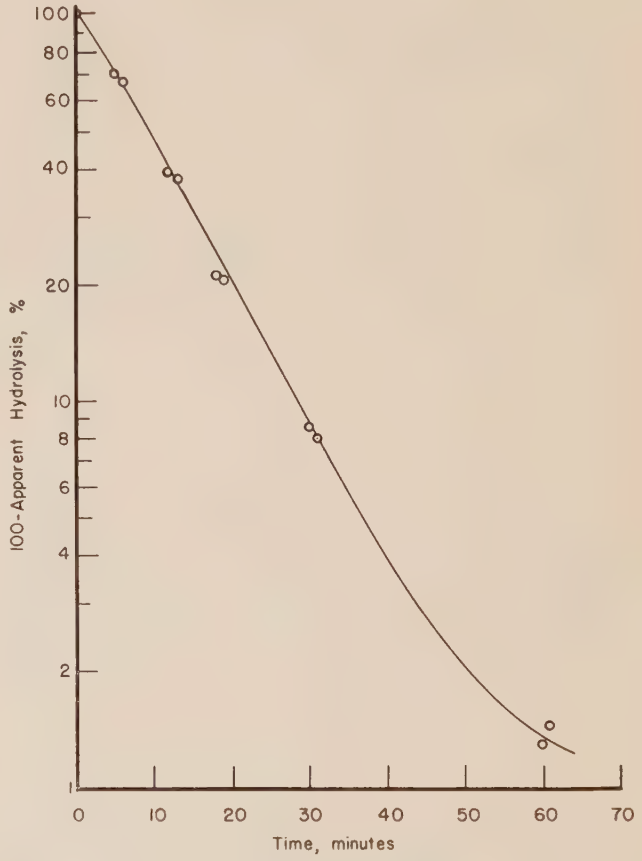

Fig. 3. Hydrolysis of birchwood crystalline xylan by 3% nitric acid

quantitatively hydrolyzed by a stepwise procedure, the insoluble residue being reactivated by autoclaving between hydrolyses. Over 99% of the material was accounted for by aldose determinations.

Attempts to obtain a preparation with reproducible reactivity were failures. There is apparently some unknown but highly important variable which affects the results. Attempts to make a colloidal dispersion were also unsuccessful. The solubility in water is apparently sufficient to enable the smaller particles to grow at the expense of the larger ones, a common phenomenon.

EFFECT OF ENZYME CONCENTRATION

The degree of hydrolysis for amorphous samples at constant time was found to vary roughly as the square

root of the concentration for less than the limiting degree of hydrolysis. This is in agreement with the findings of Voss and Butter (*2*) on their impure substrates.

ENZYMATIC HYDROLYSIS OF PURIFIED BARLEY STRAW HEMICELLULOSES

For purposes of comparison, the rate of enzymatic hydrolysis of the purified electrodialyzed barley straw hemicelluloses is shown in Fig. 2. It will be noted that, again, a "limiting" degree of hydrolysis is reached. The resistant hemicelluloses precipitate from solution. The magnitude of the "limit" depends on the original concentration of the hemicelluloses. A greater dilution gave a higher conversion despite a 36% decrease in enzyme used.

3% NITRIC ACID HYDROLYSIS OF CRYSTALLINE XYLAN

Also for purposes of comparison, the rate of hydrolysis of the crystals with 3% nitric acid at 100°C. is shown in Fig. 3. If 100 ml. of acid are used per gram of xylan, solution is complete in less than 5 minutes, although samples removed at first become cloudy again on cooling. The apparent percentage of xylan unhydrolyzed plotted differs from the true value to the extent of errors in the aldose determination and the formation of furfural from the xylose. The use of nitric acid instead of sulphuric or hydrochloric reduces but does not obviate this latter error. The semilogarithmic curve is a straight line except during the initial solution and below 2% unhydrolyzed material, where the above-mentioned errors become prominent. Thus, the reaction is a psuedo first order one and all groupings are equally easily hydrolyzed by acids. It is interesting to note that the velocity of hydrolysis by enzymes at 45°C. is of the same order as the velocity of hydrolysis by 3% nitric acid at 100°C.

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2. Voss, W., and Butter, G., *Ann.* 534: 161–204 (1938).

Received Dec. 13, 1950. A portion of a thesis submitted in partial fulfillment of the requirements of The Institute of Paper Chemistry for the Degree of Doctor of Philosophy from Lawrence College, Appleton, Wis., June, 1949. This work was carried out under the direction of William W Pigman.

Crystalline Hemicelluloses

IV. Crystalline Mannan

ALBERT P. YUNDT

The ease with which xylan could be crystallized once the basic techniques were discovered, caused speculation as to the possibility of crystallizing another hemicellulose, mannan. Mannan preparations from ivory nut and spruce pulp, though amorphous to outward appearances, have been shown to give a moderately sharp x-ray diffraction pattern (*1*). Mannan must have a strong tendency to crystallize.

IVORY NUT MANNAN "A"

Preliminary experiments were conducted on the easily isolated mannan "A" of ivory nut prepared from the chlorite "holocellulose" of commercial ivory-nut shavings essentially according to the method of Lüdtke (*2*). This material was supposedly composed entirely of mannose sugar units, but an analysis showed only 94% mannan, even after correction for hydrolysis and solubility losses. The other 6% was not identified.

Attempts to crystallize the mannan by the techniques developed for xylan (parts I and II of this series) were unsuccessful. Although highly insoluble in water at room temperature, it readily formed relatively stable supersaturated solutions of 1% concentration. Such a solution would gradually crystallize on prolonged heating at 60 to 70°C. Crystallization became slower but was incomplete after 3 days. It will be noted that 70°C. was the critical temperature at which a cooling xylan solution first showed signs of crystallization. It is postulated that thermal agitation seriously inhibits crystallization at higher temperatures and that protective hydration shells readily form at lower temperatures.

Table I. Optical Rotations of Mannans

Material	*Concentration, g./l.*	*Solvent*	$[\alpha]_D$	$[\alpha]_{436}$
Ivory nut mannan "A"	47	6% NaOH	−46°	−84°
Slash pine mannan crystals	54	6% NaOH	−41°	...
Hess and Lüdtke's spruce mannan (*1*)	..	4% NaOH	−44.6°	...
Ivory nut mannan "A"	5.2	TAPPI cupram-monium	...	303°
Slash pine mannan crystals	4.9	TAPPI cupram-monium	...	12°

The crystals, when first visible, appeared to be rod shaped, but the ends grew more rapidly than the center so that the crystals became more or less dumbbell in shape. On continued growth, the balls would more or less coalesce. The crystals showed weak but definite double refraction effects between crossed Nicols. Repeated recrystallization did not alter the habit or facility of crystallization.

SLASH PINE MANNAN

Hess and Lüdtke selectively precipitated the mannan fraction of spruce sulphite hemicelluloses as a copper complex by using a cuprammonium solution of the material 0.2 *N* in sodium hydroxide (*1*). Attempts to

Albert P. Yundt, Member TAPPI; Graduate Student, The Institute of Paper Chemistry, Appleton, Wis.; at present Research Director, Camp Manufacturing Co., Franklin, Va.

repeat their work with slash-pine holocellulose failed. The precipitate formed but was too tenuous to handle. A selective acid hydrolysis of the xylan constituents rendered them soluble in 50% ethanol (see part II) but presumably left the mannan chains undamaged and precipitable. The mannan extract was concentrated in this way and then selectively precipitated once by the method of Hess and Lüdtke. The recovered mannan was then crystallized by the methods developed for ivory nut mannan "A" with identical results with one exception. The slash pine mannan crystals were light brown instead of white. Neither repeated recrystallization nor partial crystallization altered the color. A flash bleach with chlorite removed the color but destroyed the ability of the material to crystallize.

A mannan analysis of a repeatedly recrystallized slash pine mannan fraction showed only 50% mannan. The remaining sugars were not fully identified, but appear to be D-xylose and/or D-glucose.

Optical rotational measurements are given in Table I.

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1. Hess, K., and Lüdtke, M., *Ann.* 466: 18–26 (1928).
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RECEIVED Dec. 13, 1950. A portion of a thesis submitted in partial fulfillment of the requirements of The Institute of Paper Chemistry for the Degree of Doctor of Philosophy from Lawrence College, Appleton, Wis., June, 1949. This work was carried out under the direction of William W. Pigman.

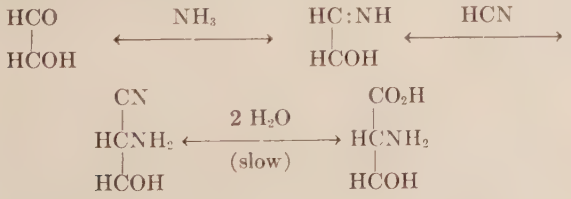
Aldose End-Group Estimation by Cyanide Consumption

ALBERT P. YUNDT

A METHOD of aldose end-group measurement was required to determine the true time rate of splitting glycosidic linkages during enzymatic hydrolysis of hemicelluloses. The usual methods of sugar analysis are either inapplicable or give large errors when applied to mixtures of simple aldoses and low molecular weight reducing polysaccharides, particularly in the presence of organic matter which is introduced with the enzyme. The hydroxylamine method of Purves and co-workers (*1*) requires titration of highly buffered solutions. Dilution and adsorption errors and pH meter drift are uncompensated.

Carbonyl compounds react with hydrogen cyanide to form cyanohydrins. Brach (*2*) allowed a sodium cyanide solution to react with aldoses at 45°C., determined the cyanide remaining by titration with silver nitrate, and compared it with a blank. The results were fair. Cyanide consumption after 12 hours was 90% of the theoretical and after 60 hours, 96%.

It was felt that this method, with modifications, showed promise. Hydrogen cyanide, the reactive material, and sodium hydroxide are produced by the hydrolysis of the sodium cyanide. Addition of ammonium nitrate to the reagent would remove the hydroxyl ions as ammonium hydroxide, whose ionization would be repressed by the excess of ammonium ions. Thus, the hydrogen cyanide concentration would be increased and, as a result, the cyanohydrin reaction would go faster and more nearly to completion. Ammonia acts as a catalyst and can enter the reaction as follows:



The end-point determination used in the silver nitrate titration was not suitable for use on turbid suspensions. Instead, a mercuric nitrate reagent was prepared containing ferric nitrate as an indicator and nitric acid. An excess of the reagent was added to the cyanide solution and the excess of mercury was titrated with standard thiocyanate to the appearance of a pink color.

PROCEDURE FOR ALDOSE ESTIMATION AS FIRST DEVISED

Approximately 0.5-millimole samples of sugar (accurately weighed) were placed in 30-ml. glass-stoppered reaction bottles and were dissolved in 5 ml. of water. Blanks of water were prepared at the same time. Five milliliters of 0.22 *M* cyanide reagent containing 200 grams per liter of ammonium nitrate were added and the solutions were well mixed. After 18 hours at room temperature, 5 ml. of 0.125 *M* mercuric nitrate containing ferric nitrate indicator and nitric acid were added and the contents were titrated to an arbitrary standard pink color with standard thiocyanate.

Testing the Procedure

The method was applied to a number of sugars as received, without special purification. The results are shown in Table I. The somewhat impure sorbitol and mannitol were analyzed for reducing sugars (as glucose) by the Munson-Walker method and the results were corrected accordingly. No immediate explanation is apparent for the anomalous cyanide consumption by

Table I. Test of Cyanide Method for Quantitative Sugars Estimation

Sugar	*Moles of cyanide consumed per mole of sugar*	
D-Glucose	1.002	0.986
D-Galactose	0.994	0.990
D-Mannose	1.000	0.994
L-Arabinose	0.998	1.008
D-Xylose	1.022	1.024
Maltose	0.948	0.962
Lactose	1.030	1.040
Cellobiose	0.966	0.980
D-Fructose	1.252	1.252
Sucrose	0.026	0.020
D-Sorbitol (corrected)	0.000	0.002
D-Mannitol (corrected)	0.003	0.001

ALBERT P. YUNDT, Member TAPPI; Graduate Student, The Institute of Paper Chemistry, Appleton, Wis.; at present, Research Director, Camp Manufacturing Co., Franklin, Va.

D-fructose. Brach tested his method with a "highly purified specimen of crystalline fructose" and reported normal results.

One-gram samples of standard cellulose consumed cyanide corresponding to degrees of polymerization of 2000 to 3000. The measured consumption was within the limits of experimental error for a blank. Tests on quantities of D-xylose from 0.025 to 0.5 millimole, using the same volume of solutions, gave cyanide consumptions within 5% of theory.

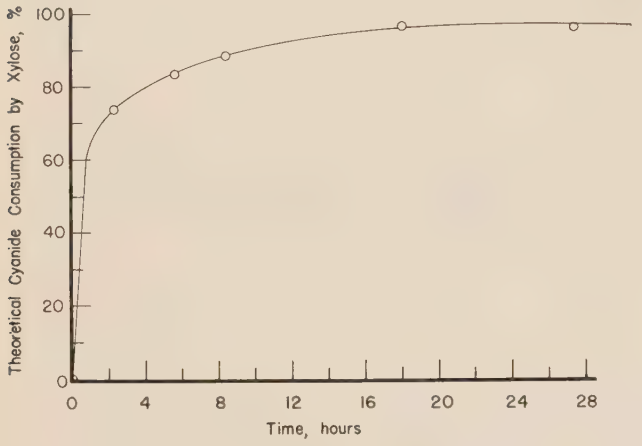


Fig. 1. Rate of cyanide consumption by xylose
Potassium cyanide, 0.11 *M;* xylose, 0.025 *M;* ammonium nitrate, 100 g./l.

The cyanide consumption of 0.25-millimole samples of xylose at varying times is shown in Table II and Fig. 1. The consumption is essentially complete after 18 hours.

Modifications of the Method

To facilitate routine operation, a pair of all-glass precision pipets of approximately 5-ml. capacity were constructed for the cyanide and mercury reagents. Each

Table II. Cyanide Consumption by 0.25 Millimole of Xylose at 23°C. in Various Times

Reaction time, hr.	*Original xylose found, %*			
	1	*2*	*3*	*Av.*
2.3	73.6	72.6	75.3	73.8
5.6	82.7	83.0	83.9	83.2
8.4	89.0	88.5	..	88.8
18.0	97.7	98.1	..	97.9
27.3	98.5	97.5	98.1	98.0

was provided with a two-way stopcock so that they could be filled by gravity from a reagent reservoir and controlled as to rate of outflow. The upper liquid level was set by a standard overflow device and the lower level was determined by a mark on a 0.5-mm. diameter capillary between the measuring bulb and the vaseline lubricated stopcock. The maximum rate of drainage was limited to 40 seconds by a constriction of the delivery tip. As the stopcock was always filled with liquid, its use introduced no error. Weighing tests with water indicated a reproducibility in delivery of ± 0.0005 ml. at constant temperature.

Despite precautions, the blanks were somewhat erratic. The trouble was traced to ferricyanide formation with the ferric nitrate indicator. The cyanide determination was changed to a mercuric nitrate electrometric titration. The procedure as developed is given below.

MODIFIED CYANIDE ALDOSE DETERMINATION

Reagents Used

Cyanide Reagent. A 0.25-*M* solution of potassium cyanide was prepared which contained 200 grams per liter of ammonium nitrate. This solution keeps about 2 weeks in a refrigerator. The pH was 8.3 when diluted 1:1 with water.

Mercuric Nitrate Reagents. A 0.10-*M* solution of mercuric nitrate was used in the pipet. For the final titration, 0.0500 $M = 0.1000\ N$ mercuric nitrate standardized against sodium thiocyanate was used. A small content of nitric acid is required to prevent precipitation of the reagent on standing.

Procedure

The sample, contained in 5 ml. of solution, was placed in a 30-ml. bottle with a mating individually lapped glass stopper. Standard taper stoppers are not satisfactory. The sample was adjusted to pH 8.3 by the addition of a predetermined volume of ammonium hydroxide. Cyanide reagent was added by a precision pipet (approximately 5 ml., precisely reproducible). The bottle was immediately stoppered and swirled to mix the solutions. For suspended samples, a rocking machine was used to keep the contents well mixed. Care was taken to avoid wetting the ground glass stopper. After 18 to 24 hours at room temperature, the bottle was opened and 0.1 *M* mercuric nitrate reagent was added by a precision pipet (approximately 5 ml., precisely reproducible). The contents were shaken well and transferred quantitatively to a 50-ml. beaker provided with a motor-driven glass stirrer, a calomel half-cell with agar bridge, and a mercury electrode. The latter was simply a platinum wire sealed into a soft glass tube and provided with an electrical connection. The short length of platinum projecting was flamed each day to white heat and freshly electroplated with mercury. The solution was titrated in its alkaline condition until the electrode emf first showed marked change. The titration was then interrupted and 20% nitric acid was added until the emf did not appreciably change on further addition of acid. The titration was then continued to an arbitrary endpoint of +50 millivolts, which falls at about the center of the voltage-titer curve inflection. Blank determinations were made using water instead of the sample and were otherwise treated as if they were unknowns. Their use compensates for variations in reagent strength. Average blank minus average titer equals net titer. Net titer, in milliliters, multiplied by the normality of the standard mercuric nitrate equals the millimoles of aldose present.

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1. Gladding, E. K., and Purves, C. B., *Paper Trade J.* 116, no. 14: 26–31 [T. S. 150–155] (April 8, 1943); *Tech. Assoc. Papers* 26: 119–124 (1943).
2. Brach, Herbert. A new method for estimating carbonyl groups in oxidized starches. Bachelor's Dissertation. Cambridge, Mass., Massachusetts Institute of Technology, 1942, 51 pp.

Received Dec. 13, 1950. A portion of a thesis submitted in partial fulfillment of the requirements of The Institute of Paper Chemistry for the Degree of Doctor of Philosophy from Lawrence College, Appleton, Wis., June, 1949. This work was carried out under the direction of William W. Pigman.

ASSOCIATION NEWS AND EVENTS

Annual Meeting Program Changes

The tentative program of the annual meeting of the Technical Association was published in the January issue of *Tappi*. Following are changes that have since been made:

Papers originally assigned to the Fundamental Research and Wet Strength Sessions have now been assigned to a Testing Session to be held at the Commodore Hotel, New York, N. Y., at 9:15 a.m., on Wednesday, February 21.

9:15 a.m. Testing (Room A)

James d'A. Clark, Research Department Engineer, Longview, Wash., *Chairman*

1. "Toward a Referee Viscosity Method for Cellulose," by A. F. Martin, Hercules Powder Co., Wilmington, Del.
2. "Moisture Determination in Paper by Oven Drying," by Geoffrey Broughton and A. K. Hobbs, Lowell Textile Institute, Lowell, Mass.
3. "Effect of Melamine Resin on Chemical Tests for Paper," by National Bureau of Standards, Washington, D. C.
4. "Characterization of Petroleum Waxes by Solvent Fractionation Methods," by F. H. MacLaren, Standard Oil Co. (Indiana), Whiting, Ind.
5. "Determination of Iron in Wood and Wood Pulp," by R. M. Kingsbury, Forest Products Laboratory, Madison, Wis. (By title).

In the Plastics Session the paper by E. K. Stilbert of the Dow Chemical Co., Midland, Mich., is entitled "The Properties of New Vinyl Chloride-Vinylidene Chloride Latex for Paper Making."

The paper "Studies of Containers Used in Export," by M. Young, Maritime Association of New York has been withdrawn from the Corrugated Containers Session and will be given at a later meeting.

In the Papermaking (Fourdrinier Session) a paper "Head Boxes and Slices," by D. R. Simonds, Beloit Iron Works, Beloit, Wis., has been added.

In the Statistics Session a paper "Groundwood Bleaching Variables," by F. R. Sheldon, Buffalo Electro-Chemical Co., Buffalo, N. Y., has been added. This paper is similar to the one given by Mr. Sheldon at the Waterville, Me., meeting of the Maine-New Hampshire Section but greater detail is given to the statistical approach to the subjects.

In the Wet Strength Session, the paper on "Factors Affecting Retention and Efficiency of Wet Strength Resins," is to be given by C. S. Maxwell of the American Cyanamid Co., Stamford, Conn.

In the Microbiological Session, the paper to be presented by Ernest Reed of Syracuse University is entitled "Microbiology of Certain Food Containers."

In the Acid Pulping Session the paper "Progress Report on the Magnesium Bisulphite Process in Operation at Longview, Wash.," by C. E. Rogers, Babcock & Wilcox Co., and Svarre Hazelquist, Weyerhaeuser Timber Co., Longview, Wash., has been withdrawn and will be presented at a later meeting.

The paper on "The Determination of Solids in Sulphite Liquor," will be presented by James H. Phillips, Babcock & Wilcox Co., Alliance, Ohio.

A paper "The Sulfox Process for Recovery of Chemicals from Acid and Neutral Sulphite Waste Liquors," by R. S. Aries and Arthur Pollak of R. S. Aries and Associates, New York, N. Y., has been added to the Acid Pulping Session.

At 2:00 p.m., on Wednesday, February 21, the Wet Strength Committee will conduct a panel discussion on "Practical Aspects of Wet Strength in Paper Manufacture."

TAPPI Coating Conference

The Coating Committee of the Technical Association of the Pulp and Paper Industry will conduct a three-day conference at the Statler Hotel, Boston, Mass., on April 18–20, 1951. Following is a tentative program:

Meetings of April 18, 1951

Symposium on Raw Stock for Coating

1. "Hanging Stock," by Thomas La Haise, Superintendent Paper Mill Division, Imperial Paper & Color Co., Plattsburg, N. Y.
2. "Off Machine Book," by A. Lewenstein, Appleton Coated Paper Co., Appleton, Wis., and M. Hausen, Bergstrom Paper Co., Neenah, Wis.
3. "On Machine Book," by J. Wilber, St. Regis Paper Co., Deferiet, N. Y.
4. "On Machine Publication," by Paul Thoma, Time, Inc., Springdale, Conn.
5. "On Machine Board (at Low Solids)," by W. C. Hunter, New Haven Pulp Board Co., New Haven, Conn.
6. "On Machine Board (at High Solids)," by speaker to be announced.

Meetings of April 19–20, 1951

1. "Hiding Power of Pigments," by J. Frankewicz and J. Senuta, Champion-International Co., Lawrence, Mass.
2. "Requirements of Litho Paper," by W. Bureau, Butler Paper Co., Chicago, Ill.
3. "Flow Properties of Clay," by C. G. Albert, Edgar Brothers Co., McIntyre, Ga.
4. "Binder Requirements for Clays of Different Particle Size," by E. K. Stilbert, Dow Chemical Co., Midland, Mich.
5. "The Trist Coating Process," by M. Trist, Trist Colloidal Products Patented, New York, N. Y. A. D. Bonanno of the Champion-International Co., is *program chairman.*

Norman I. Bearse of the Champion-International Co. will introduce the session on raw stock.

John H. Heuer of the A.P.W. Products Co., is Chairman of the Coating Committee.

The guest speaker at the Coating Conference Luncheon will be George Olmsted, President of the S. D. Warren Co., Cumberland Mills, Me.

Hotel reservations should be made personally with the management of the Statler Hotel, Boston, Mass.

Ohio Section Meeting

The Ohio Section will meet on March 18, 1951 at 6:45 p.m., at the Manchester Hotel, Middletown, Ohio. The speaker will be E. T. A. Coughlin, Chief of the Paper and Paper Products Unit of the Chemical and Plastic Section, Research and Development Branch, Military Planning Division, Office of the Army Quartermaster General, Washington, D. C.

Mr. Coughlin will discuss the details of some of the current problems on the production and procurement of paper items, with particular emphasis on those papers which are in immediate demand and of considerable volume and the use of substitutes to replace critical chemicals and materials. Some of the papers to be considered will include high wet strength map paper, sulphite writing suitable for printing, mimeograph and duplication papers, book papers such as machine finish and machine coating suitable for text booklets, manuals, and pamphlet printing, barrier papers, including coatings for waterproof, greaseproof, and MVP resistance, paperboard containers, kraft wrappings, tissues such as toweling, toilet, and napkin, paper specialties such as paper dishes and similar items.

PERSONAL MENTION

Items About New and Old Tappimen

New TAPPI Members

M. Frederick Amos, Research Chemist, Abitibi Power & Paper Co. Ltd., Sault Ste. Marie, Ont., Canada, a 1947 graduate of Acadia University with a Ph.D. degree from Purdue University in 1950.

Donald W. Bail, Shift Supervisor, Great Northern Paper Co., Millinocket, Me., a 1944 graduate of the University of Maine.

John R. Barnes, Executive Vice-President and General Manager, Moisture Register Co., Alhambra, Calif., a 1930 graduate of DePauw University.

Carroll F. Beale, Chemist, Phillips Electrical Works Ltd., Brockville, Ont., Canada, a 1933 graduate of Queens University.

Friedrich Bender, Forest Products Engineer, Forest Products Laboratory, Ottawa, Ont., Canada, a 1928 graduate of Technical University, Darmstadt, with a D.Sc. degree in 1931.

Robert E. Boehme, Assistant Chief Engineer, Panelyte Div., St. Regis Paper Co., Kalamazoo, Mich., a 1941 graduate of Michigan College of Mining & Technology, with an M.S. degree in 1946.

Giorgio Botto-Micca, Chief Chemist, S.p.A. Cartiere Burgo, Torino, Italy, a 1938 graduate of the University of Turin.

George F. Carter, Paper Technician, Paterson Parchment Paper Co., Bristol, Pa. Attended Temple University.

Hugh L. Clack, Chemist, The Dow Chemical Co., Midland, Mich., a 1942 graduate of Alma College.

James E. Cleland, Technical Director, Union Starch & Refining Co., Granite City, Ill., a 1931 graduate of Dalhousie University with an M.S. degree from the Imperial Institute of Science & Technology in 1937.

Harold V. Dearden, Managing Director, Westbury Engineering Co. Ltd., Woolfold, Bury, Lancashire, England.

Nicolas Drisch, Director of Research, Comptoir des Textiles Artificiels, Paris, France. Attended Ecole Superieure Chimie de Nancy.

Louis F. Engelhart, Chief Chemist, International Printing Ink, Elizabeth, N. J., a 1921 graduate of the University of Cincinnati.

J. A. Extrom, formerly Consultant for the Mead Sales Co. and the National Container Corp. of Wisconsin, is now associated with Chas. T. Main, Inc., Boston, Mass.

Arrigo Fedrigoni, Chemist, Cartiera Fedrigoni & Co., Verona, Italy. Attended the University of Bologna.

James W. Ferguson, Assistant Manager of Resins Div., Durez Plastics & Chemicals, Inc., North Tonawanda, N. Y., a 1931 graduate of Colgate University.

Walter M. Goodwillie, Plant Manager, United Container Co., Philadelphia, Pa. Attended the University of Illinois.

Norman L. Greenman, Development Engineer, Rogers Corp., Goodyear, Conn., a 1947 graduate of Massachusetts Institute of Technology, with an M.S. degree in 1948.

H. Ray Hall, Chemist, Allied Paper Mills, Kalamazoo, Mich., a 1941 graduate of Purdue University.

John A. Hardy, Jr., Student, University of Delaware, Newark, Del.

Robert J. Hisey, Student, Middlebury College, Middlebury, Vt.

John D. Kidd, Director and London Technical Manager, Bertrams Ltd., London, W.C.2, England, a 1928 graduate of Heriot Watt College.

Karl O. Kosk, Technical Adviser, Finnish Board Mills Association, Helsinki, Finland, a 1948 graduate of Finland Institute of Technology with an M.S. degree.

Hanna E. Krueger, Chief, Div. of Technical Service, State College of Washington, Pullman, Wash.

H. Borden Marshall, Assistant Director, Dept. of Chemistry, Ontario Research Foundation, Toronto, Ont., Canada, a 1934 graduate of McGill University with a Ph.D. degree.

Kimball A. McMullin, Assistant New England Branch Manager, Stephens-Adamson Mfg. Co., Boston, Mass., a 1939 graduate of Amherst College.

Louis M. Nourse, Librarian, St. Louis Public Library, St. Louis, Mo., a 1926 graduate of the University of Rochester; University of California Certificate in 1928, and an M.S. degree from Columbia University in 1930.

David W. Peat, Chemical Engineer, Howard Smith Paper Mills Ltd., Cornwall, Ont., Canada, a 1950 graduate of McGill University.

Karl L. Pingrey, General Superintendent, The Diamond Match Co., Ogdensburg, N. Y., a 1934 graduate of New York State College of Forestry.

Warren R. Purcell, Quality Control Manager, Brown Co., Berlin, N. H., a 1930 graduate of Worcester Polytechnic Institute, with an M.S. degree in 1932.

Warner G. Rice, General Library, University of Michigan, Ann Arbor, Mich.

George S. C. Semmens, Research Manager, Australian Paper Mfrs. Ltd., Melbourne, Victoria, Australia, a 1932 graduate of the University of Melbourne with an M.S. degree.

Ralph R. Strausbaugh, Pulp Mill Superintendent, P. H. Glatfelter Paper Co., Spring Grove, Pa.

Kenneth Thompson, Technical Div., Lowe Paper Co., Ridgefield, N. J., a 1944 graduate of Pennsylvania State College.

Jorge Ruiz Velasco, Tour Boss, Cia. Industrial de Atenquique, S. A., Atenquique, Jal., Mexico, a 1950 graduate of the University of Mexico.

John E. Ward, Research Chemist, P. H. Glatfelter Co., Spring Grove, Pa., a 1950 graduate of The Institute of Paper Chemistry with a Ph.D. degree.

Julian M. Weaver, Chief Chemist, Froehling & Robertson, Inc., Richmond, Va. Attended Virginia Military Institute, and the College of William and Mary.

Theodore E. Wooten, Assistant Purchasing Agent, Reynolds & Reynolds Co., Dayton, Ohio.

Carlos Ziegler, General Manager, Fibracel, S. A., Mexico, D. F., a 1932 graduate of Massachusetts Institute of Technology.

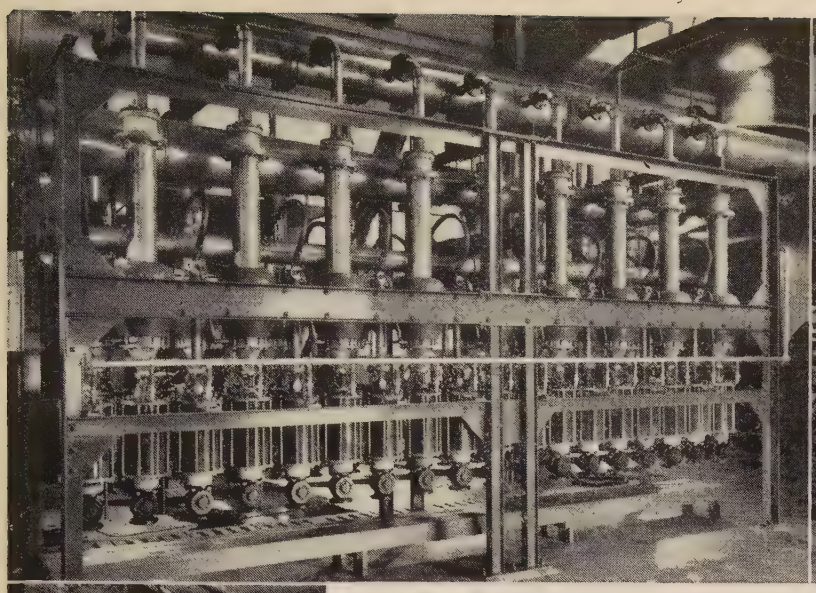
TAPPI Notes

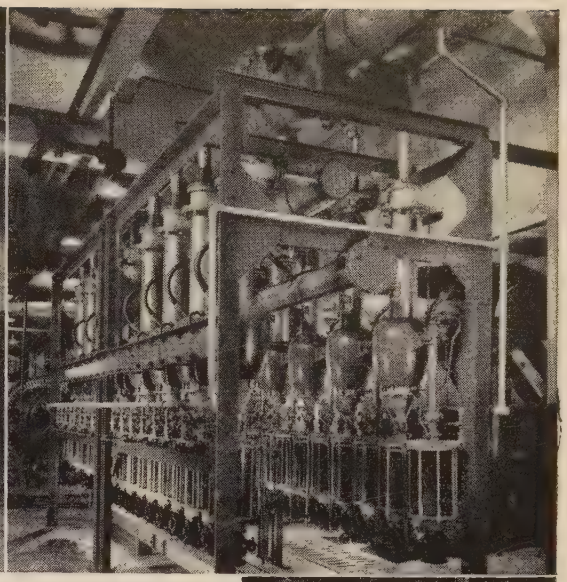
J. Rex Adams, formerly Vice-President and Technical Director of Stein, Hall & Co., Inc., is now with the Old Colony & Envelope Co., Westfield, Ore.

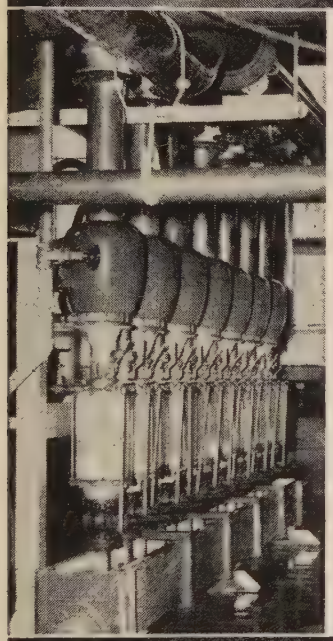
Harold R. Alley, formerly with the Army of Occupation in Japan and Okinawa, is now Manager of the Barrier and Packaging Sales Division, Charles E. King & Co., Chicago, Ill.

Carl A. Anderson, formerly of the Brown Co., Berlin, N. H., is now Asst. Chemist, Hollingsworth & Whitney Co., Waterville, Me.

Joseph E. Atchison is now Chief, Pulp and Paper Branch, Industry Division, Economic Cooperation Administration, Washington, D. C.



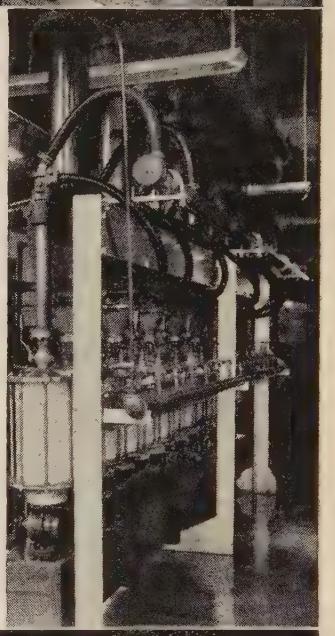


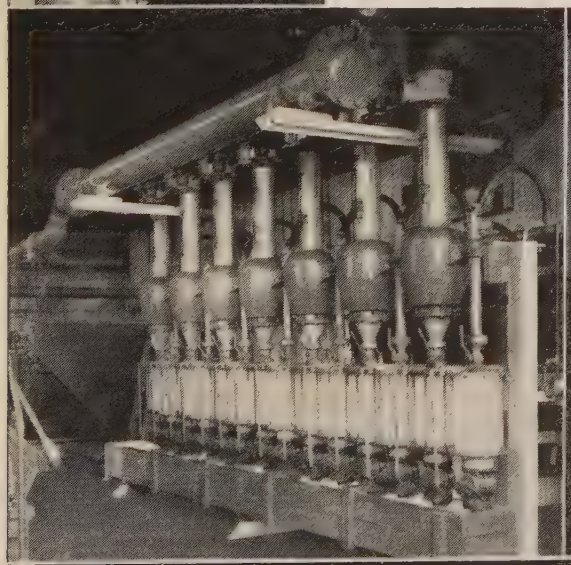


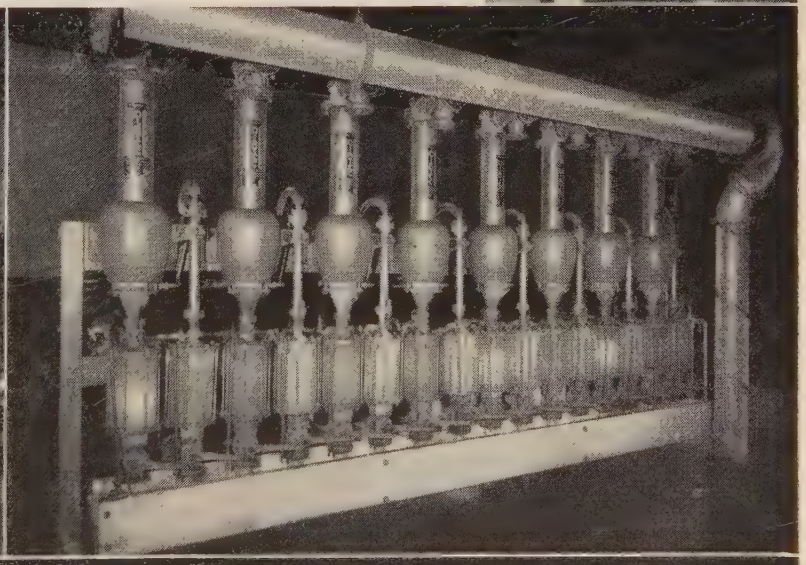
DIRTECS

Do It Better and Easier

Users everywhere like Dirtecs because they get the dirt out of the system surely and completely—because they adapt themselves so readily to every variety of dirt condition, stock and volume — and because they are so easy to operate and maintain.







BIRD MACHINE COMPANY

SOUTH WALPOLE • MASSACHUSETTS

Roy E. Bare is now Vice-President and Superintendent of the Elk Paper Mfg. Co., Childs, Md.

Nelson S. Benzing, formerly of the Champion Paper & Fibre Co., is now with the Wrenn Paper Co., Middletown, Ohio.

Bruce F. Brown is now Asst. Resident Manager, Vernon Division, Fibreboard Products, Inc., Los Angeles, Calif.

William F. Connolly, formerly with the Standard Perlite Co., is now Chemist for the Wm. H. Welsh Co., Inc., Chicago, Ill.

Robert E. E. Costello of the Abitibi Power & Paper Co., Ltd., has been transferred from Sault Ste. Marie to Toronto, Ont.

Nathan Flaxman, formerly Chemist is now Asst. Plant Manager, Peter J. Schweitzer, Inc., Elizabeth, N. J.

A. L. Hatfield of the B. F. Goodrich Chemical Co., has been transferred from Cleveland, Ohio, to Chicago, Ill.

Lawrence B. Hawes, Jr., formerly with Mason Neilan Regulator Co., is now in the Preparation Dept., Scott Paper Co., Chester, Pa.

Robert P. Kite is now Manager of the Development Dept., The Dorr Co., Stamford, Conn.

Lennart A. Lundberg, formerly Digester Cook, Weyerhaeuser Timber Co., Springfield, Ore., is now a Chemical Engineer with A. H. Lundberg, Seattle, Wash.

Carl A. Malm is now Research Chemist, British Columbia Pulp & Paper Co., Woodfibre, B. C.

Mervin E. Martin, formerly Superintendent of the Celanese Corp., of America is now with Columbia Cellulose Co., Ltd., Prince Rupert, B. C.

B. D. Millidge, formerly of Bonar & Bemis, Ltd., is now with Thomas Bonar & Co. (Canada) Ltd., Montreal, P. Q.

Sewall T. Moore is now Technical Director, A. P. W. Products Co., Albany, N. Y.

Robert G. Paris of the Kalamazoo Vegetable Parchment Co., is now Lieut. (j.g.) U.S.S. Marquette (AKA 95) c/o Fleet P. O., New York, N. Y.

Kenneth L. Rawlinson is now Paper Chemist for the Burroughs Adding Machine Co., Park Ridge, N. J.

Roy D. Rivers, formerly with the U. S. Machinery Co., is now with Thela Commercial S/A, Sao Paulo, Brazil.

Fred L. Schorken, formerly of the Central Paper Co., is now Chemical Engineer for the Oliver United Filters, Inc., Oakland, Calif.

Daniel L. Sherk, formerly of the Celanese Corp. of America, is now with Columbia Cellulose Co., Ltd., Prince Rupert, B. C.

S. C. Slifken, formerly of the Tecnifax Corp., is now with Reproduction Products Co., Franklin Park, Ill.

Bernard F. Smith, formerly of Coosa River Newsprint Co. is now Staff Engineer for Kimberly Clark Corp., Neenah, Wis.

Jerome P. Strasser, formerly of Stein, Hall & Co., is now Director of Development and Sales of Starch Specialties and Adhesives, Morningstar, Nicol, Inc., New York, N. Y.

Karl M. Thorsen is now Vice-President in charge of Production, Camp Mfg. Co., Franklin, Va.

Ward S. Yunker, formerly of the Foxboro Co., is now Sales Engineer for the Sutherland Refiner Corp., Trenton, N. J.

* * *

Members of the Technical Association are reminded that their membership will be suspended on March 31 if their 1951 membership dues are not paid by that time.

* * *

Kenneth A. Arnold, Chief of Container Research, Oswego, N. Y., has succeeded J. H. Heuer as the official corporate representative of the St. Regis Paper Co., in TAPPI.

* * *

L. O. Millard, 307 North Michigan Ave., Chicago, Ill., has succeeded H. F. R. Weber, who is on leave of absence as the official representative of the Link Belt Co., in the Technical Association.

John A. Minsen has succeeded H. J. Bartlett as the official representative of the Crane Co., Chicago, Ill., in the Technical Association.

Industry Notes

Nils G. Teren is now President of the Columbia River Paper Co., and its subsidiaries, the Oregon Pulp and Paper Co., and the Columbia River Paper Mills.

* * *

The Paper Cup and Container Institute, New York, N. Y., has announced a plan to stockpile 25 million paper cups and food containers in strategically located warehouses throughout the United States. These cups will be owned by the Institute, but will be available to duly constituted authorities in the event of disaster.

* * *

S. T. Mackenzie, head of the Philadelphia office of the Babcock & Wilcox Co., has been appointed to the newly created post of sales manager of the company with headquarters at 85 Liberty St., New York, N. Y. He is succeeded at Philadelphia by R. W. Buntin. Frank E. Hutton was recently appointed to the position of executive assistant, in charge of the sales of heat and chemical recovery equipment, digesters, alloy castings, and other process equipment for the pulp and paper industry.

* * *

The Westinghouse Electric Co. will open a plant for the manufacture of micarta at Hampton, S. C., early in 1951 and will build a new plant to make small electric motors at Union City, Ind. A new supercalender drive is announced in which both the unwind and windup are separately powered. The finished paper is wound onto a core that rests between two powered drums instead of directly onto a single motor-driven roll. With this method of windup the paper can be trimmed as it emerges from the supercalender and wound directly on the shipping core. On other drives the supercalendered roll is wound on mill cores, necessitating rewinding prior to shipment. The Company also advises that the life of the 1000-watt A-H 12 quartz mercury vapor lamp has been increased from 3000 to 4000 hours.

* * *

Paul Weller has been appointed district manager, Cincinnati office, of the Michigan Alkali Division, Wyandotte Chemical Corp. The Company also announces the construction of a new research center near the administration building in Wyandotte. It will operate under the direction of Dr. Thomas H. Vaughn, Vice-President in charge of Research. Occupancy is scheduled for 1952.

* * *

A new book on "Talc" is being distributed by Whittaker, Clark and Daniels, Inc., 260 Broadway, New York 13, N. Y. The book contains three sections dealing with uses, data, and specialties.

* * *

The Sutherland Refiner Corp., Trenton, N. J., announces a high-yield sulphate process. The process is claimed to permit a pulp mill to produce 20% more pulp from its present equipment, and gives yields in the range of 60–65% as compared to the conventional 45–48%.

* * *

The Marathon Paper Mills of Canada, Ltd., have announced that they will build a twenty-five daily ton chlorine plant at their Marathon, Ont., mill. This is the first commer-

COSTS GOING UP ?

. . . MATERIALS GETTING SCARCE

One answer is to effect savings in materials . . . lower production costs. A practical answer is the Curlator, for Curlators are accomplishing just those things. In commercial operation Curlators have increased the yield of unbleached sulphite pulp as much as 10%. In addition more than enough sulphur, coal and limestone has been saved to pay all their operating and maintenance costs. Write for more detailed information. There is no obligation.

SAVE $$$ . . . . . in Pulpwood Coal Limestone Sulphur with Curlator

Operation Proved

FIND OUT WHAT SAVINGS CURLATOR CAN MAKE FOR YOU

CURLATOR* Corporation

565 BLOSSOM ROAD · ROCHESTER 10, NEW YORK

*T.M. Reg.— Curlator Corporation, Rochester, N. Y.

CURLATOR CORPORATION
Rochester 10, N. Y.

I would like to be shown what Curlator can do for me.

NAME_____

ADDRESS _____

CITY_____ STATE_____

cial installation of De Nora mercury cells in North America. The thirty thousand ampere cells will receive d.c. power from I.T.E. Circuit Breaker Co. mechanical rectifiers. The design of the plant is such that either rayon-grade caustic or sodium sulphide can be manufactured in any proportion equivalent to the chlorine production. Hence, complete integration with pulp-mill operation will be obtained and no excess caustic will be available for sales. The project engineers for Marathon are R. B. MacMullin Associates of Niagara Falls, N. Y. The prime contractor and licensor of the De Nora process is Monsanto Chemical Company of St. Louis, Missouri, and the constructor is Leonard Construction Company of Chicago, Ill. The licensor for the sodium sulphide process is Luigi Achille, Milan, Italy.

* * *

George J. Ritter has retired from his position as chemist at the Forest Products Laboratory, Madison, Wis., after thirty years of service in fundamental research. Dr. Ritter is the author of more than sixty publications of the Forest Service. His work on holocellulose is well known to the paper industry. With this process he was able to remove any portion of the celluloses and thereby obtain new knowledge of the chemistry of wood.

He devised means of watching what happens to wood under attack by various chemicals. Thus he was able to dissect the wood cells into their tiny fibril wrappings and even smaller particles, down to the crystallites that show up only as x-ray diffraction patterns. Ultimately he was able to correlate microscopic data with x-ray finding and thereby show how the crystallites are oriented in wood. This work led to his new theory of the differential shrinking of wood.

* * *

E. I. du Pont de Nemours & Co., Inc., has announced the award of 78 postgraduate and postdoctoral fellowships to 47 universities. An authorization of $390,000 was provided by the Company for these awards. They are for the 1951–1952 academic year. This is part of the Company's plan to encourage graduate research in chemistry and other scientific fields.

* * *

Leather is being made in American tanneries with tanning extract derived from sulphite waste liquor by the Marathon Corp., Rothschild, Wis. This tanning extract replaces materials which are for the most part imported from South America. Plans are being made to develop and install during 1951 more advanced collective equipment so that more of the total spent liquor can be processed and utilized.

* * *

N. A. Ziegler of the Crane Co., Chicago, and P. H. Brace of the Westinghouse Electric Corp. have developed a process for making hardened stainless steel. The metal is subjected to a hard freeze and a cold squeeze and becomes one of the hardest metals known. By this treatment stainless steel overcomes some of its natural disadvantages and becomes available for uses in which wear resistance and high strength are required.

* * *

The International Paper Co., Southern Kraft Division, has issued three interesting bulletins as part of its effort to promote good forest management, fire prevention and tree farming. One is the Jackie Davis comic book which was placed in the hands of 1,500,000 school children in the South, 55,000 copies of the supplementary "Suggestions to Teachers," and "A Report to the People of the South 'If we all work together we can lick fire.' "

* * *

The Diamond Alkali Co., 300 Union Commerce Bldg., Cleveland 14, Ohio, has issued a Chlorine Handbook which may be obtained from the Company.

Jerome P. Strasser

Frank E. Hutton

Jerome P. Strasser, former Vice-President of Stein, Hall & Co., has joined Morningstar, Nicol, Inc., 630 West 51st Street, New York 19, N. Y., to direct the development and sale of starch specialties and adhesives for the paper industry. The expansion of Morningstar, Nicol sales and technical staffs for the papermaking and converting industry continues a development program that began in 1931 when its subsidiary Paisley Products, Inc., was established. In 1939 the Aroostoocrat Potato Products Inc., plant was built at Houlton, Me., to produce potato starch.

* * *

The Wisconsin Pulp and Paper Research League recently investigated the feasibility of burning waste liquor from the ammonia-base operation of the Wausau Paper Mills. The liquor was concentrated at the League's pilot plant in Appleton and the burning tests were made in a furnace at the Interlake Mill of the Consolidated Water Power and Paper Co., in Appleton. The tests showed that the ammonium liquor can be burned as fuel, that ammonia odor is not likely to be a problem, and that fly-ash is apparently less difficult than fly-ash from the burning of calcium-base liquor. The boiler efficiency obtained from this test was considerably below the rated capacity of the equipment. The League evaporator plant encountered difficulties in the course of boiling out water from the ammonia-base liquor. Evaporator surfaces unexpectedly fouled with carbon because of the greater viscosity of ammonia-base liquor. Until this evaporator difficulty is overcome, further burning tests on this material must be postponed.

* * *

The Materials Handling Exposition will be held in the International Amphitheatre, Chicago, Ill., on April 30 to May 4, 1951. There will be ten acres of exhibits.

* * *

Paul Scallan, Lloyd G. Schenk and C. Fred Asker have been elected vice-presidents of Perkins-Goodwin Co., 30 Rockefeller Plaza, New York, N. Y.

* * *

Hoberg Paper Mills have announced plans to build a $1,250,000 plant here for evaporating and burning spent sulphite liquor. Expectation is that this will reduce sulphite pollution from Hoberg's pulp mill by approximately 40%. The plans and specifications have been submitted to the Committee on Water Pollution and the State Board of Health for approval. Following is a statement by J. M. Conway, President:

"As soon as the state authorities approve the project, Hoberg will undertake construction. We shall be ready to place orders for equipment and to let contracts whenever they tell us we can go ahead.

Reproduction of an advertisement of the *first* paper mill in America.
from an engraving made in Philo. about 1825 by P.C. Hamm.

Courtesy The Historical Society of Pennsylvania

According to reliable historians, the first paper mill in America was established in 1690 near Germantown, now part of Philadelphia, Pa., by William Rittenhouse, William Bradford, and others.

Too bad Bradford and Rittenhouse couldn't use

PENNSALT LIQUID CHLORINE

Credit these paper pioneers with a job well done—in view of the conditions under which they worked! Without chlorine, and other essential chemicals, the excellent processed papers of today were far beyond achievement of these early manufacturers.

Many pulp and paper mills throughout the nation now rely on Pennsalt Liquid Chlorine for

Bleaching of wood pulp and other papermaking fibers. Slime Control in pulp and paper mill process water.

And these mills use Pennsalt chlorine with confidence. For just as Bradford and Rittenhouse were pioneers in the paper manufacturing industry of this country, so has Pennsalt pioneered in the production, handling, storage and shipment of chlorine.

In fact, the first tank car of liquid chlorine ever to travel American rails left Pennsalt's Wyandotte plant in the year 1909. Today, thousands of tons are shipped yearly in tank cars, both single and multi-unit, and in cylinders . . . from conveniently located plants in

Portland, Ore. Tacoma, Wash.
Wyandotte, Mich.

Backing up its chlorine production, Pennsalt continues through intensive research to help improve chemical processes within the pulp and paper industry. Competent technical service from a Pennsalt representative is at all times available upon request.

Write today for information on prices and delivery.
In the West: Pennsylvania Salt Manufacturing Company of Washington, Tacoma, Washington; Portland, Oregon; Los Angeles, California.
In the East: Pennsylvania Salt Manufacturing Company, Philadelphia 7, Pa.

PENN SALT

liquid chlorine

OTHER PENNSALT PRODUCTS: Caustic Soda • Anhydrous Ammonia • Bleaching Powder • Perchloron® • Hydrogen Peroxide • Sodium Arsenite • Sodium Chlorate • Sodium Hypochlorite • Forest Insecticide Sprays

PENNSALT DISTRICT OFFICES: Philadelphia • Tacoma, Wash. • Portland, Ore. • Los Angeles • Bryan, Texas • Chicago • Cincinnati • Wyandotte, Mich. • Paterson, N. J. • Pittsburgh • St. Louis • Appleton, Wis.

Progressive Chemistry for over a Century

"A year ago at a hearing in Green Bay we told the Committee that all known methods of treating spent sulphite liquor were in an experimental stage, and that in our opinion a satisfactory solution to this problem would not be forthcoming until some type of full scale plant would be put into operation.

"These statements are also true today. Considerably more is known now than a year ago about treating spent liquor to reduce stream pollution, but a great many points remain to be cleared up and the subject remains experimental. A full-scale plant, with the opportunities that this will afford for development work, seems to us to be the only possible way to work out a completely practical solution."

At the December, 1949, hearing Mr. Conway asked for one year in which to complete plans, and agreed to build once his company's plans were approved. The state pollution authorities accordingly issued an order directing Hoberg to submit plans and specifications by Dec. 31, 1950. Today's action was in compliance with that order.

The same state order instructed Hoberg to complete by the end of 1950 installation of fiber recovery equipment to meet state standards of fiber loss to the stream. In today's report to the official bodies, the company pointed out that every division of its pulp and paper operations is now fully complying with the requirements. By installing new and improved equipment and by rearranging its water system for maximum reuse and recirculation, Hoberg has substantially cut its fiber loss and decreased its use of water per ton of paper produced.

It has been widely rumored that Hoberg would choose the yeast treatment process, by which torula yeast is propagated in sulphite liquor to consume the wood sugars. Actually Hoberg engineers and technical men designed a yeast process plant twice as large as the commercial scale pilot plant built at Rhinelander by cooperative action of Wisconsin sulphite pulp mills. "Experience has convinced us that the construction of a yeast plant is impractical at this time because of the lack of secondary treatment for the effluent and uncertainty of the degree of pollution reduction which would result," explains the company's report to the Committee on Water Pollution. Undoubtedly also contributing to the decision were the difficulties that the Rhinelander plant has encountered recently in disposing of yeast produced in the course of its treatment process.

"Construction and operation of the evaporate and burn plant will give us an opportunity for a thorough evaluation of this process," Mr. Conway indicated. "The proposed construction will create the opportunity for us eventually to further develop the evaporate and burn process, or to pursue the yeast process with secondary treatment of its effluent, or to use both processes as future developments may make advisable. Hoberg wishes to reduce its pollution of the Fox River to the greatest practical extent, just as rapidly as science makes methods available."

Northern Paper Mills, Green Bay, Wis., have announced that it will construct at an estimated cost of $1,500,000 a plant to reduce sulphite pollution of the Fox river. First moves to get under way will be taken by the company just as soon as state authorities approve its plans, the statement declared.

The company pointed out in its release that the new plant is the culmination of many years of research to find a workable solution to the problem of disposing of spent sulphite liquor. Pulp manufacturers have long maintained that sulphite liquor presents the most difficult task in reducing pollution from industry wastes.

At a public hearing on Dec. 22, 1949 Northern testified that it had researched, in cooperation with other Wisconsin mills, for years on this problem without getting a final answer. However, according to its testimony at that time, a ray of hope had developed in the research, so that a method practical to install and operate might develop reasonably soon.

In his company's report to the state bodies, Milan Boex, President stated, "The time allotted to Northern by the Committee's order did not permit full exploration of all of the conditions that may be encountered in commercial operations. Indications are that an installation should enable Northern to reduce the pollutional effects from spent sulphite liquor by 40 to 45%."

"The process of evaporating much of the water from spent sulphite liquor to concentrate its content of dissolved wood solids and then burning it as industrial fuel was selected as best adapted to Northern's situation after investigation of three other promising processes."

In orders issued on Feb. 21, 1950, state authorities requested Northern and Hoberg Paper Mills, both of Green Bay, as well as Consolidated Water Power and Paper Company's Interlake Mill at Appleton, to submit by Dec. 31, 1950, detailed plans and specifications to reduce their pollution of the Fox River to an acceptable minimum. The state's order further stipulated that upon official acceptance and approval of these plans, construction is to be completed by Dec. 31, 1951.

The estimated $1,500,000 cost to complete the proposed installation covered by Northern Paper Mills' plans at today's cost levels does not include a building. The company already has a building which with some modifications can be made adequate to house the proposed equipment for collecting and evaporating the spent liquor.

In the formal report, which is signed by Mr. Boex, Northern agreed to start to negotiate and place orders for required equipment promptly after its plans are officially approved.

Pointing out to the state authorities that stainless steel is essential in handling spent sulphite liquor, the report likewise called to their attention that emergency defense demands have already made stainless steel extremely difficult to obtain for civilian use, with every indication that this scarcity will become even more acute. "If Northern eventually should find itself unable to obtain necessary materials, the Company will ask the Committee to take up with federal materials control authorities the public importance of pollution reduction and assist in obtaining the requisite priorities so that any plans which the Committee accepts can be carried through with the required materials," Boex notified the state bodies concerned.

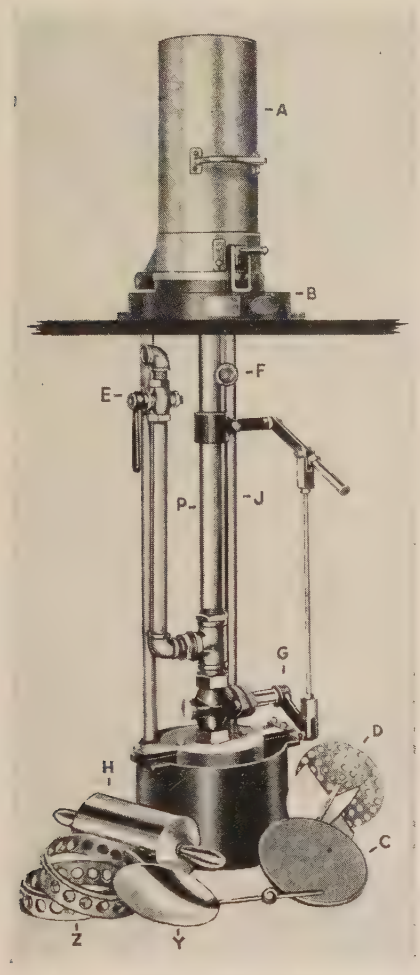
Northern emphasized that it sincerely wishes to carry through its plans to completion. But to guard against any possibility of finding itself forced by some United States federal defense regulation to violate some part of a Wisconsin state order, Northern formally requested the Committee on Water Pollution and the State Board of Health to modify their order of Feb. 21, 1950, so that the time limit for completing construction will be automatically extended if for defense or other reasons beyond the company's control it should find itself unable to obtain equipment required for its proposed treatment plant.

Accompanying Boex's report, Northern supplied to the state authorities a set of large scale engineering drawings of the proposed installation, a flow chart showing how the spent sulphite liquor will be handled, a description of the process and full specifications. Besides the treatment plant proper these documents disclose, the company is changing some of its pulping practices in order to make the treatment process feasible.

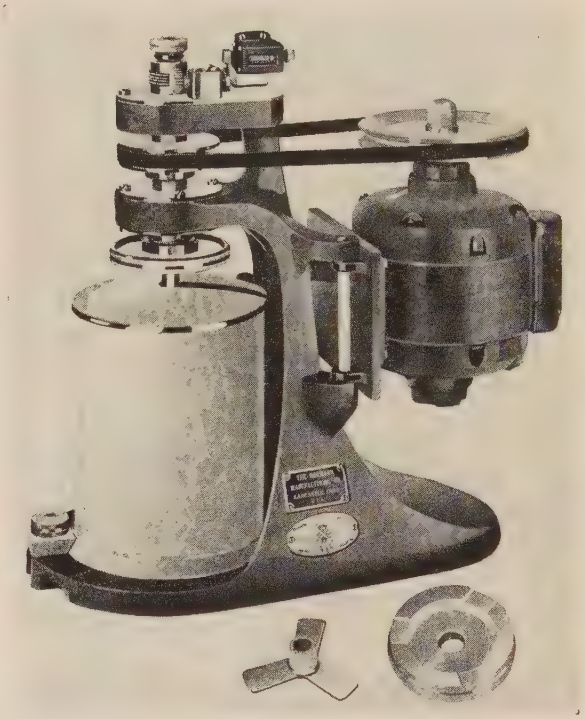
Instead of placing water in a blow pit as a pad before blowing in the contents of a digester, as at present, the mill will pad the blow pit with liquor from a previous cook. This change will decrease the dilution of the incoming spent liquor which eventually must be boiled down to greater concentration. It is expected that liquor collected for processing will average about 10% solids in solution. Instead of discharging spent liquor to the sewer from the blow pits, each blow pit will be equipped to pump the liquor to a 10,000-gal. stainless steel padding tank, through screens, and then to a 100,000-gal.

TAPPI STANDARD

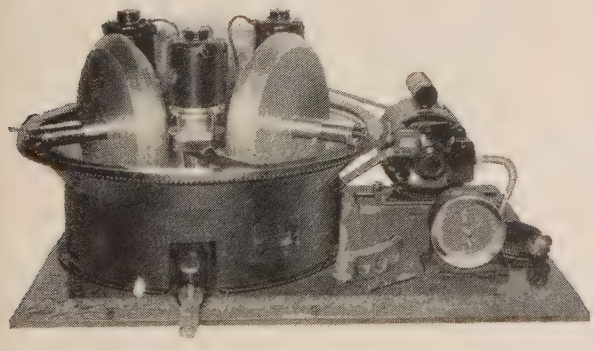
PULP TESTING—SHEET MAKING APPARATUS



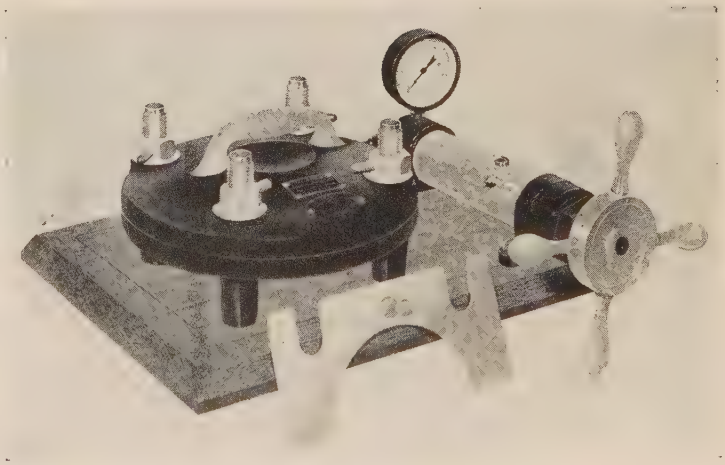
*SHEET MACHINE—DRYER RINGS—DISCS



*DISINTEGRATOR



LABORATORY KOLLERGANG BEATER



*PUMP AND PRESS

NEW CATALOGUE AND QUOTATIONS GLADLY FURNISHED ON REQUEST

THE HERMANN MANUFACTURING CO.

MFRS. ALSO: HERMANN IMPROVED CLAFLIN CONTINUOUS BEATER & REFINER

LANCASTER, OHIO

wooden storage tank. This capacity is sufficient to store liquor from five digester blows, or about 12 hours collection.

The collected 10% spent liquor will pass from storage to a five-vapor body quadruple effect evaporator of the reversing type designed to prevent excessive lime scaling. Here the liquor will be evaporated to 52% concentration. The 52% material will in turn pass through a two vapor body concentrator of reversing type for further reduction to a solids content between 60 and 65% so that it can be burned in the power plant boiler through atomizing burners. The concentration operation involves handling 101,000 lb. of 10% liquor hourly through evaporation to end up with 19,400 lb. of heavy concentrated liquor. The concentrate will be stored at 200° F. in a supply tank adjacent to the boiler plant.

Fly ash from burning the spent liquor is high in calcium content. It is about 50% finer than fly ash which results from burning coal, and presents an accordingly more difficult problem to collect and dispose of. Plans for fly ash collectors on the mill's two existing boilers will be submitted to the City of Green Bay for approval under its air pollution ordinance. Because the fly ash is so fine, special handling will be required for hauling it from the plant after it is collected.

Collection and evaporation equipment will be housed in a 40 by 140 foot three-story brick building of fire-resistant construction. This structure is already built, but requires one major modification. The proposed evaporator equipment would be too high for the present roof level, and so the roof must be raised 14 ft. to accommodate these units. The operating floor of the evaporation equipment will be about 25 feet above the ground floor.

* * *

Our steady approach to a war economy reminds us of some of the developments in the industry during World War II. We are sorry that we do not know the source of this information but presume that it was the War Production Board.

Alcohol—from waste liquor and wood waste, both potable and nonpotable. Can replace alcohol made from grain.

Binder Twine—to replace sisal and henequin in agricultural binder twine of which about 250 million pounds are used annually on American farms.

Bottle Corks—made of cardboard faced with plastic.

Braided Twisted Paper—to replace steel strapping in baling clothing, cotton, etc. and used in place of string in many applications.

Corrugated Paper Boxes for Air Freight—lighter than wood; water and vaporproof for shipping by air perishables such as fruits, vegetables, and fish, as well as all types of commodities.

Creping Wadding—for packing fragile materials.

Fiber Cans and Tubes—to replace metal cans and tubes.

Fiber Cones, Ribbon Blocks, Bobbins, Spools, etc.—for textile plants.

Fluorescent Paper—for night operations, should be widely used in the theatrical field.

Impregnated Creped Cellulose Wadding and Paper—a rubberlike material, impervious to gasoline, oils, organic solvents, and gases, with stretch characteristics similar to those of pliable leather.

Impregnated Plastic Paper—for plywood surfacing, luggage, nonslip flooring and table tops.

Impregnated Plastic Paper—for molding into flat or curved shapes for doors, floors, megaphones, etc.

Laminated Paper Plastics—having a tensile strength comparable, pound for pound, with aluminum, which can be molded to desired shapes at the low pressures and temperatures available on equipment now used for making plywood. Will be important in the construction of homes, furniture, and in many industrial applications by the automotive, electrical equipment, radio, and other industries.

Laminated Asphalt Impregnated Wood Fibers—for heat insulation for buildings.

Mold Retardant Wrapper—for bread, cheese, and butter, and other foods. The wrapper is impregnated with a chemical which does not affect the flavor but retards the formation of mold.

Packaging of Compressed Food—as an example, one pound soup bricks which will feed 36 people. Also includes dried eggs, dried milk, 11 fruits and vegetables, 7 grain products, beef, pork and dry sausage, dry salt fish, and tea.

Packaging Frozen Foods—containers will be water vaporproof, greaseproof, and made of material which will be odorless, tasteless, and noncorrosive.

Packaging and Storage of Dehydrated Vegetables—protects the quality of the contents under severe conditions of export shipment and storage; will confine gases or vapors without leakage for long periods: product must be protected from moisture absorption at temperatures occasionally as high as 130° F., and as low as −15°.

Paper Automobile License Plates

Paper Base Plastics—used in England for railroad baggage cars and in Canada for printing plates.

Paper Blankets—lighter than wool and just as warm.

Paper Clothes—cheaper than cloth.

Paper Curtains and Draperies—cannot be washed but can be dusted and cleaned.

Paper Insulation—for insulating ceilings, walls, and floors of buildings.

Paper Oil Containers—to replace tin for lubricating oil supplies by filling stations, etc.

Paper Parachutes—for dropping flares and supplies.

Paper Raincoats—used by commandos in some of their raids.

Paper Sand Bags—a multiwall paper bag with asphalt laminated outer and inner sheets, to be used as sand bags. A California oil refinery has had these exposed to the weather for one year without apparent deterioration.

Multiwall Paper Bags—for potatoes, peanuts, salt, corn, dry chemicals, dog food, plaster, cement, fertilizers, sugar, etc.

Paper Shipping Sacks—to resist climatic changes, exposure to poison gases and fumes, insect infestation, rain, salt water, sleet, snow, ice, blistering heat, freezing weather and tropical humidity.

Paper Stretchers—the San Francisco Dept. of Public Health is using paper stretchers. A paper stretcher on continuous 24-hour service is averaging 5 days' use before being discarded. The cost is 40 cents against $4.50 for canvas stretchers.

Paper Tarpaulins

Paper Tea Bags—very strong when wet, permeable to liquids, impassive to solids.

Paper Vests

Paper Wingtips—for airplanes; will hold loads 40% greater than the design load for similar structure made of aluminum. The British Mosquito plane, one of the world's lightest, fastest, and most useful planes is built of layers of paper and plywood. The Lancaster Bomber contains over 1500 paper components. Gliders have been made of paper.

A Paper and Wood Combination is being used as flooring in cargo planes.

Paper Wiping Cloths—to replace textile cloths; used extensively by the Navy.

Paperboard Clothes Hanger

Safety Paper—to prevent falsification of records.

Surgical Dressings—absorbent wadding, substitute material for rubber sheets, dental napkins.

Antitarnish Tissue—free from any acid or sulphur compounds injurious to articles of highly polished metal and leaded glass.

Crepe Tissue Emergency Bandages—used on battlefields and in hospitals immediately behind the lines.

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liquid phenylmercuric acetate

POSITIVE SLIME CONTROL

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Pitch and Asphalt Dispersant

Low cost Pitch and Asphalt control based upon sound chemical principles. P-A-D dissolves pitch or asphalt out of your system.

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There are a few remaining unassigned territories for exclusive distribution of our products available to the most promising interests.

W. A. CLEARY CORPORATION

NEW BRUNSWICK, N. J.

Hospital Bed Sheet Tissue—a multi-ply tissue paper, the bottom sheet of which is water repellent.

Lens Tissue—to clean lenses replacing that formerly made only in Japan.

One-Time Carbonizing Tissue—used by the armed forces for making up order forms.

Parachute Tissue—used for parachutes carrying light weights such as flares.

Tracing Tissue—a special high grade white tissue made for tracings and charts for blueprinting and other reproduction purposes.

Twisted Tissue Bags—woven in more open mesh than jute for onions and citrus fruit.

Tall Oil—a by-product in the manufacture of paper has been found to be a good wetting agent in the manufacture of natural and synthetic rubber. Also used in the manufacture of soap, paints, varnishes, cutting oils, and wetting agents.

V-Board—developed for packaging shipments to the armed forces. It is so waterproof that V-containers are thrown overboard near Pacific islands and are washed ashore by the tide.

Water and Greaseproof Paper—for wrapping and packaging.

Water Repellent Packaging Tape—has a waterproof bond and is impervious to extremes of temperature and of mold. Under test the tape remained sealed to a V-type carton after being submerged in water for 70 days.

Waterproof Coated Tag—now used in place of metal for the time cards on letter boxes. Will find other applications where durable notices are desired.

Waterproof Envelopes

Wet-Strength Paper—loses but little strength in wetting, developed as map paper for the armed forces. Used for bags, tags, fruit wraps, paper towels, photo papers, and other miscellaneous applications.

Wet-Strength Tags—as substitutes for cloth tags made with the strength resins used in the map papers made for the armed forces.

Woven Paper Burlap—A wide and close mesh product of twisted kraft paper to replace burlap for floor covering backing and bags. It is made in water repellent mildew-resistant, and flame-resistant forms.

It has been estimated that one cord of pulpwood converted into the basic part of smokeless powder is used to fire 90,000 bullets in a Garand rifle or 420 shells from a 105-mm. gun, or 24 shells from 16-inch guns. It will also produce 4200 weatherproof packages to carry life-saving blood plasma, 3336 containers for first-aid kits with emergency battle dressing and sulfa tablets, 16,444 hospital waddings for field treatments and emergency operations, 1500 fiber parachutes for bombing flares or for food supplies, 1440 antitank mine covers, 2148 watertight jackets for 37-mm. shells, 800 warm vests for aviators in high altitudes, 1560 containers for K rations, 900 tough multiwall commando bags for bulk foods and supplies, 1200 yards of ordnance wraps to protect airplane engines from rust, 6120 military maps, 72 protective rings for 1000-pound bombs, and 1800 grommets for 5-inch shells.

* * *

The following items dealing with the shortages of copper and sulphur are statements issued by E. W. Tinker, Executive Secretary of the American Paper & Pulp Association. They were prepared by the Critical Materials Committee of the A.P.P.A. and have been presented with oral arguments to the National Production Authority.

Woven Wire Cloth Supply

The paper and board industry is the sixth largest industry in the United States in value of product, according to the U. S. Bureau of the Census.

A wartime listing of paper uses shows paper utilized as a substitute for metal foil, wood, glass, silk, canvas, leather, ceramics, hemp, rubber, felt, sawdust, plastics, cork, mica, shellac, plaster, lead, skins, tin, slate, straw, ink, gold, silver. The most amazing part of the list is the hundreds of uses where paper alone will suffice, and the fact that although paper is a substitute for so many things, there is listed not one single substitute for paper itself.

Paper today is not only printing and writing paper, it is the basic commodity without which no person, no business, no government can exist in any mode remotely resembling what we know as modern civilization. It is the only commodity without which every commercial enterprise, every educational institution, and every government function would immediately cease.

Paper is money, postage stamps, letters, railroad tickets, construction blueprints, law books, bills of lading, electrical insulation, government orders, business records, food packaging, shipping containers, grocery bags, newspapers and magazines. It is building material, navigational charts, towels, telegrams, pictures, checks, lamp shades, calendars, telephone directories, stocks, bonds, contracts. It is a thousand other things so omnipresent and so essential that they are completely taken for granted.

In other words, the availability of paper in great variety, in large quantities every day, under any and all circumstances, in peace or in war, in boom or in depression, is an absolute necessity to this country. Its essentiality to any country is in direct proportion to the state of civilization and industrialization of that country.

We are presenting herewith the pattern of paper consumption by end use which was developed by the War Production Board Pulp Allocation Office for 1944, a period when the United States was fighting on many fronts all over the world.

The following table shows the volume of paper and board consumed for eight end-use categories, and percentages for these. The first two categories depend upon the size of the armed forces. The activities of the next two—Communication, Transportation, Public Service; and Agricultural Products and Drugs—are greatly increased during wartime. Construction in 1944 was almost entirely related to wartime activity. The result was that in 1944 these war-related activities took 72.1% of the production.

Copper Requirements for the Paper and Pulp Industry

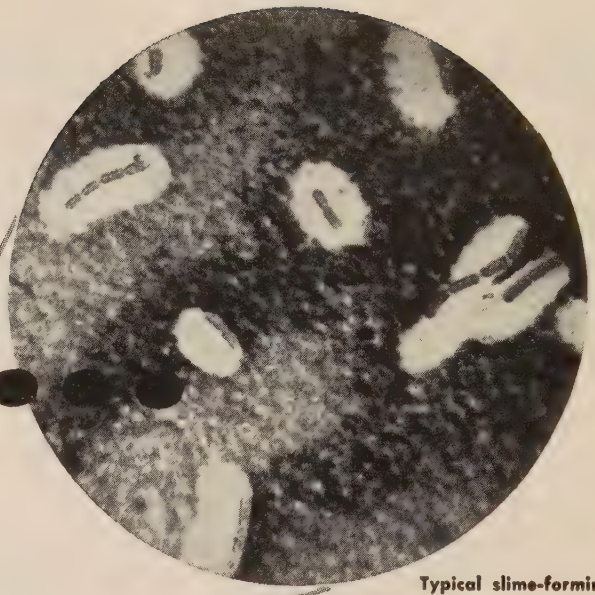
The manufacture of pulp and paper requires the use of both chemical and mechanical processes in the course of the reduction of fibrous raw material to pulp and the further processing of pulp into paper. A large part of the process is carried on in the presence of chemical solutions, and the digesters, pipes, valves, and parts of the machinery which come in contact with the material being processed require the use of a substance that resists destructive corrosion.

Bronze and copper have been used in much of the pulp and paper mill piping and in many machine parts.

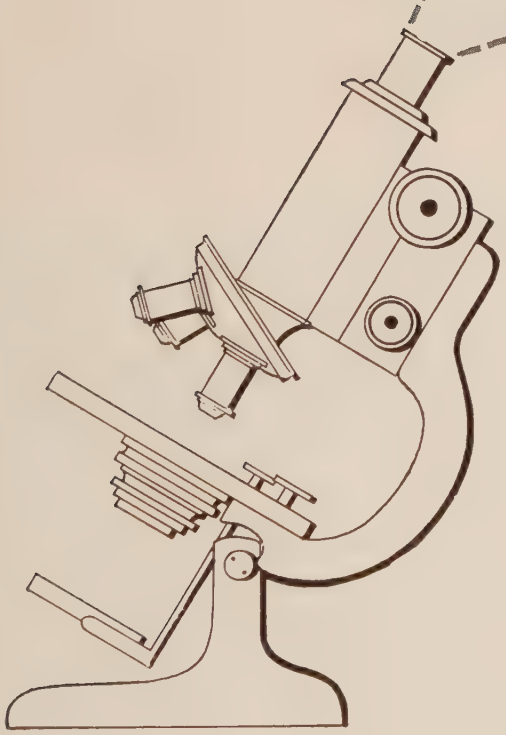
By long odds the most important machinery part is copper or bronze woven wire cloth. This is used in many applications where water is separated from the fiber being carried in solution. The principal application is on the paper machine, either as a fourdrinier wire or as a cylinder mold cover.

	Wartime end-use pattern, paper and board	
	Tonnage 1944 (thousand tons)	*Total 1944, %*
Government and F.E.A.	2,217	13.2
Defense plants	4,268	25.4
Communication, transportation, public service	1,797	10.7
Agricultural products and drugs	2,856	17.0
Construction	974	5.8
Manufacturers of civilian products	2,335	13.9
Retail and household	1,008	6.0
Miscellaneous	1,344	8.0
Total	16,799	100.0

kill these pests...



Typical slime-forming organisms



with CHLORINATION by W&T

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Wallace & Tiernan Chlorinators are particularly effective in slime control work because they are especially designed from over 35 years' experience to give low cost, efficient performance.

For a survey on the chlorination needs of your mill without obligation, call your nearest W & T Representative now and start your plant on the road to slime free operation.

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CHLORINE AND CHEMICAL CONTROL EQUIPMENT
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I-36

Material	Requirements in terms of pounds of metal per ton of paper and board (over-all averages)	Absolute tonnage based on an annual production of 26,500,000 tons of paper and board
Copper	0.22	2942
Tin	0.012	162
Zinc	0.018	237
Monel metal	0.005	63
Stainless steel	0.0125	166

Other important uses are on the cylinder washers, savealls, wet machines, filters, deckers, and extraction rolls.

It is certain that without this woven wire cloth material, production of paper and board is impossible. A curtailment of the supply means a corresponding curtailment of production by the paper and board industry.

This wire cloth is made by specialists in its production, who make it up to the exact specifications of the paper and board manufacturers.

The requirements of scarce materials for paper mill wire cloths are comparatively small.

The above table of requirements is based on estimates furnished us by the Fourdrinier Wire Manufacturers Association.

Other Copper and Brass Savings

It is demonstrable that substantial savings of copper are possible in the pulp and paper industry without stopping production. But these savings can be made only in uses of copper other than in woven wires, which are absolutely essential to the full operation of the industry. A committee of the industry is working on this problem and will report its findings at an early date.

Scrap Returnable

Much of the copper used by pulp, paper and board mills will be returned in the form of scrap. There will, however, be a difference in the *rate of return* from woven wire cloth and the *rate of return* from other copper uses. Woven wire cloth is consumed very rapidly, and approximately 85% of it returns quickly to the stream of copper scrap supplies.

This fact is most important from the standpoint that the net drain on the copper supply by the industry is reduced to the almost infinitesimal figure of $437\frac{1}{4}$ tons of copper to make 26,500,000 tons of paper and board. This means that over 60,000 tons of paper and board are produced for every ton of copper consumed in the form of woven wire cloth! A 20% cut in wire cloth would save 87 tons of copper and lose 5,300,000 tons of paper and board production.

Recommendations

W.P.B. Order L 209 was issued in November, 1942, as an inventory control order for copper woven wire cloth. That it was workable and satisfactory to all concerned is evidenced by the fact that it was one of the very few W.P.B. orders that remained unchanged in the stress and strain of the strenuous war years that followed. It was carefully worked out and even under the tight Controlled Materials Plan, enough copper was allotted to this end use to satisfy the requirements of the pulp, paper, and board industry.

Because copper woven wire cloth is essential to the maintenance of the production of pulp, paper and board, we unanimously recommend that adequate supplies of copper, tin, zinc, monel metal, and stainless steel be made available to the producers of woven wire cloth so that they can supply the pulp, paper and board industry on the same basis as in World War II. If controls are deemed necessary, we recommend that Regulation 2 be clarified by an order similar in tenor and effect to W.P.B. L 209.

Excerpts from W.P.B. Order L 209, Nov. 2, 1942

Paragraph 3094.1 General Limitation Order L 209:

(a) Definitions (for the purposes of this order):

(1) "Producer" means any individual, partnership, association, business, trust, corporation, or any form of enterprise whatsoever, whether incorporated or not, engaged in producing pulp, paper or paperboard.

(2) "Wire cloth" includes any type of metal wire containing copper, nickel, chrome, or alloys thereof which has been woven into wire cloth for use in the manufacturing processes employed to produce pulp, paper or paperboard.

(3) "Paper mill machine unit" means those industrial machine units (of the type listed in Part I of Schedule A attached) employed in the manufacturing processes for the production of pulp, paper or paperboard.

(b) Restrictions on acceptance of deliveries of wire cloth: No producer shall accept any delivery of wire cloth for any paper mill machine unit if acceptance thereof would increase his total inventory (including wire cloth held for his account) of such wire cloth for that paper mill machine unit in excess of the numbers and kinds indicated for such type of paper mill machine unit in Part II of Schedule A attached.

(c) Restriction on orders and inventories of wire cloth: No producer shall place any order for wire cloth or enter into any other arrangement for the fabrication of wire cloth without specifying approximate delivery date; and no producer shall specify any delivery date earlier than such producer may reasonably expect, on the basis of average wire life experience, to be able to accept delivery of such wire cloth without violating the provisions of paragraph (b) of this order.

SCHEDULE A

Part I—Paper Mill Machine Unit	Part II—Authorized Wire Cloth Inventory
Fourdrinier machine	4 (complete wires)
Cylinder machine and washers:	
One-cylinder machine	2 backings, 2 facings per machine
Two-cylinder machine	2 backings, 2 facings per machine
Three-cylinder machine	2 backings, 2 facings per machine
Four-cylinder machine	3 backings, 3 facings per machine
Five-cylinder machine	3 backings, 3 facings per machine
Six-cylinder machine	3 backings, 3 facings per machine
Seven-cylinder machine	4 backings, 4 facings per machine
Eight-cylinder machine	4 backings, 4 facings per machine
Nine-cylinder machine	4 backings, 4 facings per machine
Saveall	2 backings, 2 facings per unit
Wet machine	2 backings, 2 facings per unit
Deckers	2 backings, 2 facings per unit
Filters	2 backings, 2 facings per unit
Extractor rolls	3 backings, 3 facings per unit

Sulphur Requirements for Wood Pulp Production

Importance of Wood Pulp in the National Economy

Wood pulp is a basic raw material for the production of paper, paperboard, rayon, smokeless powder for military explosives, plastics, photographic supplies, etc.

Of the total economic wealth created annually by American industry, more than *one tenth* is derived from values added in the manufacture of wood pulp and its major dependent products. These values, contingent upon an adequate supply of wood pulp, are essential to the maintenance of a virile national economy.

Pulp products are essential to the production, distribution or use of *all* materials of war and of *all* essential civilian supplies. The dependence of *all* segments of the economy upon pulp products makes it vital, from the standpoint of national defense, to maintain the supply of this basic commodity.

In two world wars, the United States and her Allies have been seriously handicapped by shortages of pulp and pulp products. These shortages were attributable in part to sharp increases in wartime demand and in part to the fact that

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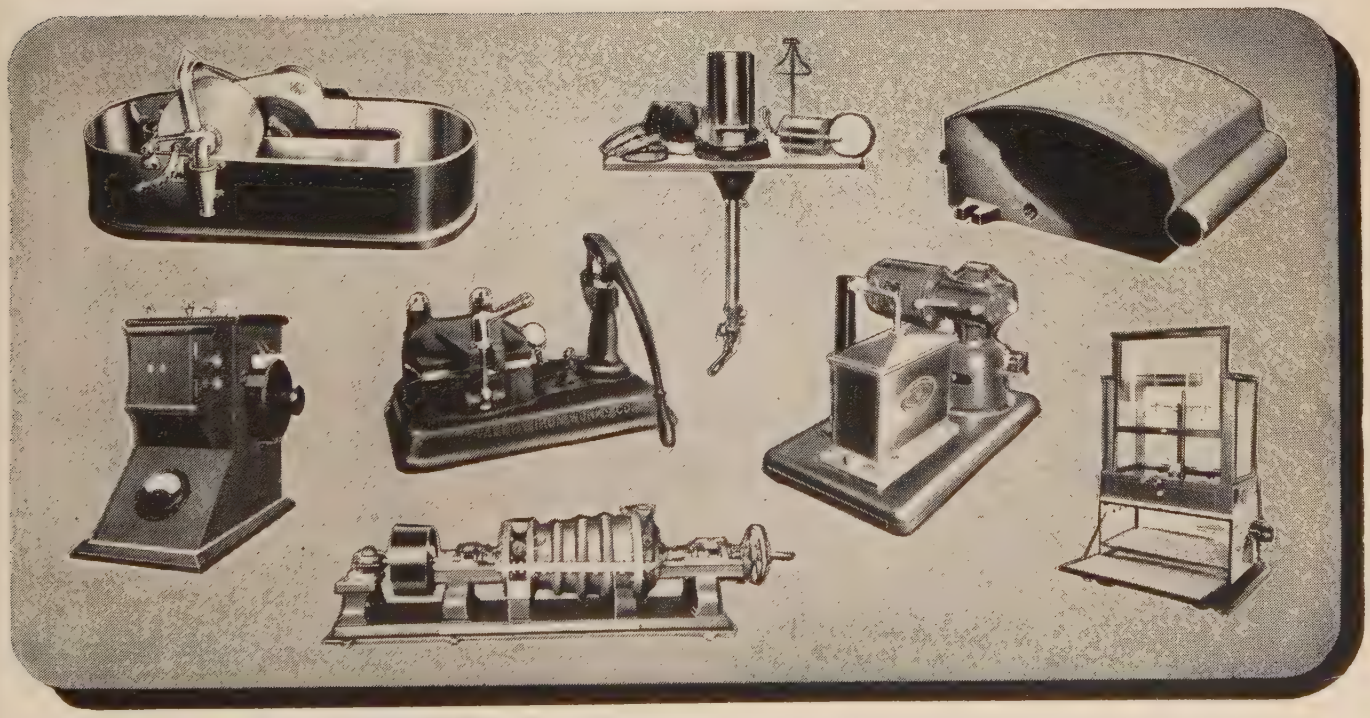

VALLEY *Laboratory Equipment*

Scandinavia, a major supply source for wood pulp, was cut off from world trade. In the event of another world war, Scandinavian pulp, representing 20% of the world's supply, would again be unavailable to the Western Powers. North America must again be prepared to meet the essential wartime needs of the Atlantic Pact Countries for vital pulp products.

At the present time, even with Scandinavian pulp moving freely in world trade, there is an acute world shortage of wood pulp. The countries of Western Europe, through O.E.E.C., are already trying desperately to meet essential needs for pulp and pulp products by increased deliveries from the American continent; despite general improvement in Europe's economic indices, her pulp and paper indices have not yet equalled prewar levels.

If pulp and pulp products are to be continuously available in volume adequate to meet defense needs, then domestic facilities for the production of wood pulp must be maintained and kept operative. If facilities, at the outset of the emergency period, are allowed for any reason to become idle, it may well become virtually impossible to reactivate them.

Without adequate supplies of sulphur, the domestic pulp industry will be faced almost immediately with the necessity of seriously curtailing wood pulp production.

Curtailment of pulp production to conserve sulphur can only mean, under present conditions, that world shortages of wood pulp will assume alarming proportions, that the servicing of basic military and civilian needs will be seriously hampered, and that the success of the mobilization program of the Western Powers will be jeopardized.

There is no substitute for sulphur in the manufacture of sulphite pulp.

Sulphur Requirements for Pulp Production

Based on a survey completed by the United States Pulp Producers Association, Inc., in December, 1950, the United States pulp industry's consumption of sulphur in 1950 totaled 436,477 long tons. Because of increased capacity and increased demands generated by the mobilization program, this industry's sulphur requirement in 1951 will total 481,920 long tons.

If consumption of sulphur in pulp production is restricted in 1951 to 85% of 1950 consumption, then the supply of sulphur available for consumption this year will be 110,000 tons, or 23% short of meeting the pulp industry's 1951 needs.

Since sulphur is essential to pulp production, a sulphur shortage of a magnitude of 23%, would result in a serious curtailment of domestic pulp supply at a time when there are desperate domestic and international pressures for an increased supply of wood pulp to meet essential world needs.

Overseas' Exports of Sulphur

Despite the fact that essential domestic production is threatened with serious curtailment because of an inadequate sulphur supply, and despite the fact that our known domestic reserves of sulphur will be largely liquidated in a relatively few years, sulphur is still being shipped overseas in substantial volume.

Overseas' export commitments for the first quarter of 1951 are equal to 16% of our total prospective domestic supply of sulphur for the quarter.

To the extent that sulphur exports in subsequent quarters of 1951 can be reduced, the problem of North American supply will be proportionately eased. An embargo on overseas' exports would completely solve, for the short-term at least, our North American supply problem.

Europe should be put on notice immediately that exports of sulphur from North America will be embargoed at the earliest practicable date, and that steps must be taken at once by those countries that have been dependent upon the United States to obtain their requirements from other sources.

Relative Essentiality of Use

Most of the industries competing for the available sulphur supply are basic industries of prime essentiality.

Nevertheless, it is possible that some of these industries can accept substantial cuts in sulphur supply, during the emergency period, without serious consequences to the domestic economy.

The production of fertilizer, for example, requires an estimated 21% of the total domestic consumption of elemental sulphur and an estimated 34% of the total domestic consumption of sulphuric acid. Sulphur consumption in fertilizer production has increased sharply in recent years. Two factors have been largely responsible for this increase:

1. Marshall Plan aid, which has resulted in a 303% increase in the rate of exports since 1945.
2. Farm aid, which has included free fertilizer for growers of certain farm products.

Sulphur consumption in the fertilizer industry might be reduced without injury to the national economy or the mobilization program by increasing the supply of fertilizers produced from raw materials other than sulphur.

There is no substitute for sulphur in the manufacture of sulphite pulp.

Conservation of Use

The outlook for sulphur supply, for the long-term as well as the short-term, makes it urgent for all domestic industries to begin at once programs designed to insure maximum practicable efficiency in the use of sulphur.

The government should initiate at once an effective conservation and reclamation program in which all American industries should be urged to cooperate.

Until a few months ago, this industry had no indication of a shortage of sulphur. Improvements in operating efficiency cannot be accomplished overnight. Every effort should be made to maintain the flow of sulphur to essential uses in volume adequate to maintain essential production until such time as improved efficiencies resulting from improvements permit a reduction in sulphur deliveries.

An abrupt curtailment of sulphur supply can only result in an irretrievable loss of pulp supply and in inestimable damage to our national war potential.

Conclusions

Recommendations presented in this report may be summarized as follows:

1. The government should consider carefully the economic consequences of permitting continued overseas' exports of sulphur and the economic justification for initiating, at the earliest practicable date, a complete embargo on overseas' exports.

2. Limitation orders should be directed first at those domestic uses of sulphur that can be curtailed with minimum injury to the over-all domestic economy and with the least possible damage to the major objectives of the mobilization program.

3. A joint government and industry conservation and reclamation program, designed to insure an efficient use and recovery of sulphur, should be initiated at the earliest practicable date.

4. Until such time as an effective conservation and reclamation program can become operative, sulphur deliveries to essential industries should be maintained at a level adequate to insure the continued production of basic industrial raw materials needed in the mobilization program.

Wood pulp is a basic industrial raw material of prime essentiality. In view of present and prospective world shortages of this commodity, any policy that results in a curtailment of supply would appear, from past experience, to be ill-advised.

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OBITUARIES

Ralph A. Hayward

Ralph Allington Hayward, President of the Technical Association of the Pulp and Paper Industry during 1942 and 1943 died on Jan. 11, 1951, at the University Hospital, Ann Arbor, Mich., from thrombosis of basilar artery. He was buried in Kalamazoo, Mich., on Jan. 15, 1951.

Ralph Allington Hayward, President of TAPPI (1942–1943)

Mr. Hayward was born in St. Clair, Mich., on April 16, 1893, the son of Capt. George W. Hayward and Susan W. (Lowe) Hayward. He attended public school in Ashtabula, Ohio, and graduated as a chemical engineer from the University of Michigan in 1917. On Oct. 6, 1917 he married Marion W. MacGregor.

After graduating from the University of Michigan he became resident engineer for the Spanish River Pulp and Paper Co., at Espanola, Ont. In August of the same year he joined the Ordnance Dept., of the U. S. Army serving as District Metallurgist in the Rochester, N. Y., District. In 1919 he joined the consulting engineering firm of the R. B. Wolf Co., and in this capacity was resident manager and engineer of Fraser Ltd., Edmundston, N. B., consulting engineer for the Miami Paper Co., Dayton, Ohio, and manager of the Newton Falls Paper Co., Newton Falls, N. Y.

In 1923 he returned to the University of Michigan as Asst. Professor of Chemical Engineering. In 1924 he joined the Kalamazoo Vegetable Parchment Co., Kalamazoo, Mich., as general manager. In 1936 he became President of the Company. In addition he was Chairman of the Board of the Appleford Paper Products Ltd., Hamilton, Ont., and the Harvey Paper Products Co., Sturgis, Mich.

Mr. Hayward was a Regent of the University of Michigan, Trustee of Kalamazoo College and member of the Michigan State Highway Advisory Board, Board of Directors, Michigan Bell Telephone Co., Board of Directors, First National Bank of Kalamazoo, Board of Directors, Sutherland Paper Co., and the Board of Directors, Federal Home Loan Bank of Indianapolis, Ind.

He is survived by his mother, his son John M. Hayward, Parchment, Mich., his brother Dr. Roy G. Hayward, and sisters Mrs. G. H. Gotshall and Mrs. W. H. Olney.

Carl Ernst Apelgren

Carl Ernst Apelgren, Technical Director of the Kraft Paper Division of the St. Regis Paper Co., died of coronary thrombosis at his home in Oswego, N. Y., on Dec. 26, 1950.

Mr. Apelgren was born in Hernosand, Sweden, on July 29, 1895, and graduated as a chemical engineer from the Swedish State Technical College in 1916, following which, from 1916 to 1923 he worked as a chemist for the Nensjo Sulphate Mill, the Storvik Sulphite Mill, the Kalix Sulphate Mill, the Kvarnsveden Paper Mill, and the Sandarne Sulphate Mill.

In 1923 he came to the United States where he became a chemist for the American Writing Paper Co., Holyoke, Mass. In 1925 he joined the Filer Fiber Co., technical staff at Filer City, Mich., and in 1928 he joined the Taggert Corp., now the St. Regis Paper Co.

EMPLOYMENT SERVICE

Positions Open

P94-50. Established organization engaged in the development of specialty products for the paper industry desires research man with extensive organic and physical chemistry background. Ph.D. desirable but not essential. Give details of education, experience, and salary requirements.

P98-51. Sales engineer. Immediate opening for technically qualified man, familiar with alkaline pulping and all pulp and paper mill processes, to head promotion work at home office of leading manufacturer of industrial instruments. Occasional traveling. Location: New England. Graduate chemical or mechanical engineer preferred. Position permanent. Salary. Good company to work for; congenial associates. Send full particulars.

P99-51. Chemical engineer wanted for development work relating to manufacture of wood fiber products. Work involves process design, plant and pilot plant operations. Up to five years' experience preferred. Location: Upper Midwest. Submit details of training and experience, salary requirements, and references.

P100-51. Wood technologist with masters' or bachelors' degree required by development department of company manufacturing a wide variety of wood fiber products. Should have a knowledge of mathematics through calculus and be interested in the mechanical, thermal, and acoustical properties of materials. Location: Minnesota.

P101-51. Experienced paper machinery designer, with special knowledge of reels, winders, and cutters. Excellent opportunity with well-established company.

P102-51. Chemist or chemical engineer for sales and service work selling adhesives to coated paper trade. Location: Middle West.

P103-51. Chief chemist. Kraft mill located in South. Preferably man in late twenties with about five years' experience, including supervision.

P104-51. University graduate with knowledge of theoretical and practical aspects of cooking and bleaching kraft pulp wanted by Midwestern paper mill making fine papers.

Positions Wanted

E101-51. Chemical engineer. 30. Family. Five years' diversified pulp and paper experience in control, engineering, and development in newsprint, sulphite, soda, groundwood, and waste paper. Presently employed. Desires position in production or production development. Prefer location in West or Midwest.

E105-51. Engineer. Graduate civil and mechanical engineer having 30 years' extensive pulp and paper mill experience in design and manufacture of kraft, sulphite, and groundwood grades, desires to make a connection with a company as an administrative or operating executive.

E106-51. Mill superintendent with proved ability in conventional or Yankee fourdrinier mill manufacturing light or heavy weight kraft or sulphite specialty grades desires new connections.

E107-51. Technical director. Twenty years' experience in production and quality control of kraft, sulphite, and groundwood pulps and papers. Best of references.

E108-51. Mill engineer—available soon; knowledge of fourdrinier specialty and cylinder machines; technical graduate; sulphite pulp and paper mill design and plant engineering experience.

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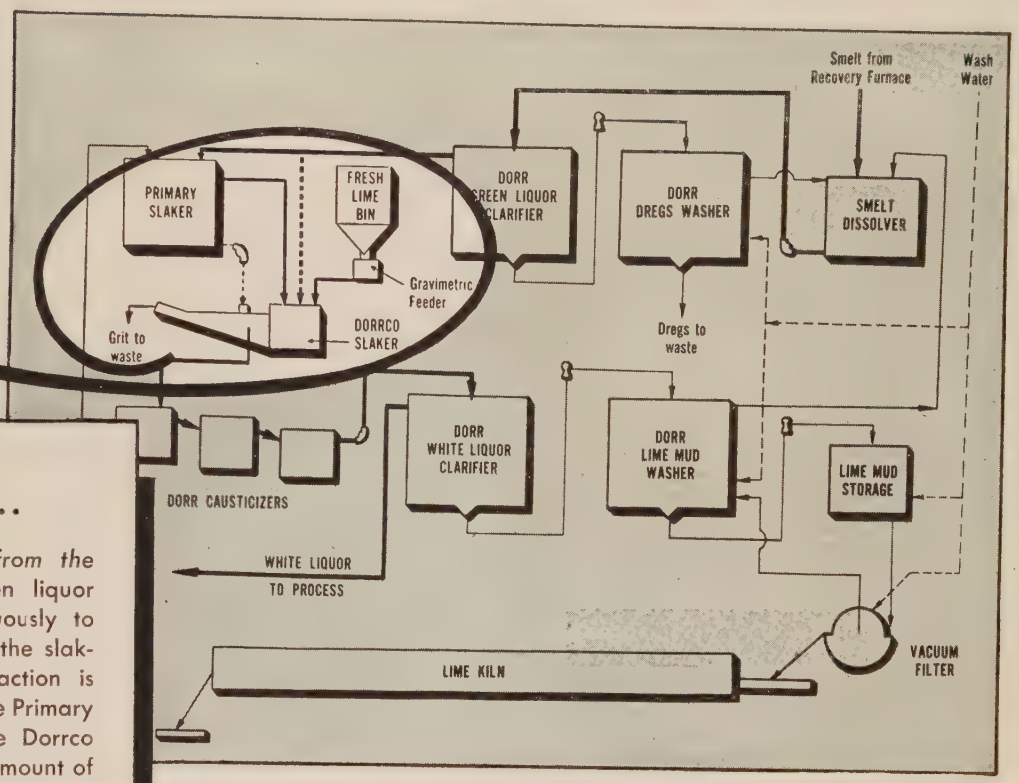
The slaking station of the Dorr Continuous Recausticizing System . . . standard of the industry . . . has been redesigned to provide an even higher degree of operating efficiency.



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Reburned lime, *direct from the kiln,* and clarified green liquor are now added continuously to a Primary Slaker where the slaking and recausticizing action is started. Overflow from the Primary is fed by gravity to the Dorrco Slaker and a measured amount of makeup lime is added. Slaking is completed in the Dorrco, which produces a clean milk of lime and a washed, separated grit. The remainder of the flowsheet is the standard Dorr System.

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LOCAL SECTION ACTIVITIES

Reports of Meetings, Personnel and Events

Pacific

A discussion of "Printing and Print Papers" by an outstanding group of men in the paper manufacturing and printing fields constituted the program of the regular meeting of the Pacific Section at Camas, Wash., Nov. 7, 1950. The technical program was arranged by E. H. Nunn, Technical Supervisor, Crown Zellerbach Corp., West Linn, Ore. who served as moderator. Other members of the panel were Maurice Adler, California Ink Co., Berkeley, Calif.; Milton Bell, Abbott, Kerns & Bell, Portland, Ore.; G. R. Cranor, Oregon Pulp & Paper Co., Salem, Ore.; and B. H. Wright, Crown Zellerbach Corp., West Linn, Ore.

A résumé of the papers follows.

Ink Problems in Printing, by Maurice Adler, California Ink Co.

There are three fundamental types of printing—Intaglio, Planographic, and Letterpress.

The inks used for all three basic methods of printing have two things in common. Each has a pigment to give the visual characteristics of the ink, whether it be black or white, or any other color. Each type of ink also has a vehicle to carry the pigment from the ink fountain on the press through the various steps in the ink distribution system until it reaches the medium on which it is to be printed—the ultimate goal of all printing inks.

During the process of printing, both ink and paper must not only have characteristics in relationship to each other, but together they also must have a relationship with respect to the conditions of printing, particularly the make and speed of the presses on which they are being used. For example, it is easy to conceive of a paper and ink being used on a press running say a thousand feet per minute with complete satisfaction for hours. Yet, the same paper and the same ink, if used on a press running about one hundred feet per minute, would cause all kinds of trouble—so much so that both paper and ink would have to be changed before the printing of the job could progress satisfactorily. However, changing of the ink alone might take care of the situation.

The two major components of printed matter go hand in hand. Of course, we also have ink problems where no paper problem is present, but they are not of as much interest to you paper technicians.

A problem that is ever present in the field of newspaper printing is what is commonly known as "Second Impression Offset." In newspaper printing, first one side of the sheet is printed, and in a fraction of a second the printed side is pressed tightly against the impression cylinder while the opposite side is being printed. The ink being moist causes a slight transfer from the paper to the impression cylinder. This causes an accumulation of ink on the impression cylinder as the run progresses. Soon there is enough accumulated on this second-impression cylinder to offset back on to the paper, causing a smudge or dirty appearance over the entire side of the paper that was printed first, and it usually shows much worse on halftone cuts. This is what is called "Second Impression Offset." You can see it every day in the paper.

There is a definite solution to this problem but it is a question of cost.

A problem on which the entire Ink Industry constantly labors is the development of brilliant colors on brown kraft wrapping paper, and brown kraft container. This type of problem can easily be illustrated by a request we recently had from a large sugar company. They submitted to us a display of colors they were using and a set of new artist paint swatches which they wished us to duplicate not only on their white carton and bleach sulphite, but also on brown kraft and brown container board.

(The artist's swatches and color matches, and commercial matches were shown.)

Every once in a while a firm that has been for years printing a job by the Letterpress method decides to change to the Lithograph Offset method, commonly referred to merely as "Offset." As you know, the thickness of the ink film applied to the paper by the Offset process is from one third to one half the thickness when applied by the Letterpress process. This calls for inks with a high-color concentration, and sometimes it is very difficult to attain this without running into press difficulties. Here is a set of cartons, one having been printed by Letterpress—the other by Offset. You will note that the one printed Offset gives better detail in the vignettes, and a generally better quality of printing.

With respect to ink, two very important properties of a paper stock are its wettability and capillarity. It was the difference in these properties that caused the trouble just mentioned about picking of the enamel stock.

I have here a collection of proofs that were made with the same ink on various types of papers. When these proofs were printed, an attempt was made to have the same amount of ink on the roller and plate for each proof. That is, the ink was put in the fountain, and each of the paper stocks were printed, and when each print was made, the amount of ink used was replaced by the ink ductor roller. We can say, then, that the same amount of ink was applied to each type of paper. The appearance of each, however, shows that there is a great difference in the way the ink wets the paper, and the way the capillary action causes the ink to penetrate into the spaces between the fibers.

This brings us to a point where the entire ink industry has bumped up against a problem of supplying inks to be used on machine-coated papers. This type of stock has the peculiar property of being very thirsty for the vehicle, and inks printed on it are robbed of much of their vehicle to the point where the stock is satisfied. If the ink does not have sufficient vehicle to satisfy the stock, all of it is removed from the ink, leaving nothing but the pigment on the surface of the paper. This results in the ink chalking—that is, the pigment has no vehicle left to bind it to the surface of the paper. Inks normally formulated for use on enamel papers do not have sufficient vehicle to satisfy the thirst of machine-coated papers, and will usually chalk. If proper adjustment is made, however, such as the addition of enough vehicle, binder, and dryer, such inks will get by very nicely. Unfortunately, so many shops have all kinds of paper stocks in their inventory. Too often there is an oversight, when ordering ink, to specify accurately the type of stock which is to be used. Once in a while there is a misunderstanding, and an ink is made for enamel stock when actually it is to be used on machine-coated stock. Then somebody is unhappy. It is occurrences like this that bring home to us very strongly the importance of close collaboration between the paper supplier, the ink supplier, and the consumer.

Problems Arising in the Printing of Paper on an Offset Press, by Milton Bell, Abbott, Kearns and Bell

"Of all the troubles lithographers are heir to, none can be more annoying or more crippling to production than the antics of paper. Paper can stretch, shrink, curl, develop wavy or tight edges, wrinkle on the press, become electrified, and defy all efforts to make it behave in a respectable manner."*

In most cases, paper misbehaves under the influence of atmospheric humidity.

In our plant we like to have stock with a moisture content of 7%. This is usually 3 or 4% more moisture than paper contains when it comes off the paper machine. In order to bring it up to 7% moisture content it, therefore, must be especially conditioned before being shipped to the printer.

Dry stock moved to a moist atmosphere stretches. Stock with a high-moisture content, when moved to a low humidity pressroom, will shrink. If one side of the stock moistens or dries more than the other, the sheet will curl. Covering the top and sides of stock piles corrects this by preventing the absorption or escape of moisture. Curling also occurs in running too much moisture on the press. This can of course, be corrected by reducing the dampening water to a minimum. A third cause of curling results from cutting stock too close to the reel. Nothing much can be done about this except to reject the stock.

A pile of dry paper standing in a humid atmosphere will absorb moisture of the edges, while the center of the pile is unaffected. Wavy edges result. This works conversely with high-moisture content paper in a dry room. The edges shrink as moisture content shrinks—the interior of the pile remains moist. When the edges tighten up, the paper sags in the center.

The humidity of the pressroom, the moisture content of the paper and the number of times the sheets must go through the press all have a bearing on the problem of static. There is,

* "Paper and Humidity in the Pressroom," Lithographic Technical Foundation.

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of course, considerable friction as the sheets pass over a number of steel rollers on the feeder and the press. Dry sheets tend to acquire and hold a static electricity charge in a very stubborn fashion. We have had presses tied up for as long as 5 days fighting static.

Undersize sheets, oversize sheets, partial sheets, lint, and other foreign material occasionally found in skids of paper create additional problems on an offset press.

Quality Control of Printing Papers, by G. R. Cranor, Oregon Pulp & Paper Co.

Any discussion of paper for printing should include paper, ink, and the various processes by which the two are brought together into a finished product. To do this in any detail is not intended by or within the scope of the writer.

Until comparatively recent years the papermaker has considered his sheet a finished product. He judged his paper on such factors as furnish, weight, and various physical tests that indicated strength and other characteristics.

In the following sections several of these characteristics will be briefly discussed with no effort made to arrange them in order of importance to printing.

Physical Strength. The strength of a sheet of paper is probably not as important to printing as it is to the ultimate end use of the printed product. The sheet, of course, must be able to withstand the stresses placed upon it by the press but of greater importance is the relation to the binding operation such as folding and stitching. The paper must have strength so that pages of books, magazines, etc. will not crack and fall out.

Surface Strength. A characteristic of very great importance to the printer is surface strength. This is especially so as the speed of the presses are increased, the use of higher pressures, and employment of inks of greater tack. Surface strength is the resistance to pick or the lifting of the surface of the paper during printing.

The test in general use for determining surface strength is the wax pick test. Waxes of various adhesive strength are manufactured and given a numerical rating.

The test consists of melting a series of graded waxes and sticking them to the surface of the sheet. After they have cooled for 15 minutes, they are lifted vertically with a quick jerk. The paper has the highest wax test that has not broken or injured the surface of the paper.

The proof press is also recommended for determining pick by using inks that are going to be used in the printing process.

The pick tests, so far developed, do not indicate one very important factor of picking that causes considerable trouble and that is the fine picking over a wide area that will fill up a plate and yet not be evident from the ordinary pick tests. Printers, at times, have found that paper with a high pick rating will do this. The test area is too small in relation to a full-sized printing area to indicate this characteristic. The proof press, or the roller test probably give the most reliable results in this regard.

The formation of a sheet of paper is important to the papermaker but it is probably of greater importance to the printer. This characteristic is basic for printing paper because many of the printing factors are dependent on good formation.

There are many printing troubles that are directly caused by poor formation or by the results obtained by the papermaker trying to smooth the hills and valleys by calendering.

Smoothness which is closely related to formation is a very important factor in printing papers. It governs many other factors in printing. The smoother the sheet the better or more uniform is the contact with the printing plate.

The smoothness of paper is generally measured by an air-flow method wherein a definite volume of air under a constant pressure is allowed to pass between the surface of the sheet and a reference surface. The rate at which the volume of air passes between the two surfaces is given as the smoothness of the surface. The Bekk, the Williams, and the Gurley S.P.S. are instruments used in this measurement and vary only in the method used to apply the same principle.

Softness or Compressibility of paper is its ability to compress and conform to the shape of the printing plate under pressure. This softness in a sheet of paper is of great advantage if other characteristics are not effected. If the sheet has some resilience, under pressure, the sheet will crush to such an extent that all parts of the printing surface are able to make contact with the paper and give a better impression, thus overcoming, to some degree, unevenness of the surface.

Softness is determined by measuring the compressibility of the paper. Bekk has developed two instruments for measuring this factor, one under static pressure and the other under dynamic pressure. The first measures the thickness under increasing loads. The second uses a pendulum with a rounded head that can be inked and dropped from a definite height onto the sheet and the rebound and size of the inked spot measured. The softer the paper the less rebound and the larger the ink spot. The Gurley S.P.S. tester uses the air-flow method. The bottom ring has found steel pins 0.002 inch above the surface that allows a definite amount of air to pass between the reference plates. These steel pins will penetrate a sheet placed between the plates under pressure and thus reduce the air flow. The softer the paper the more the flow is reduced and the farther the test reading is from the standard air flow.

Porosity or the ease by which air passes through paper has been considered a measure of the ability of paper to absorb oil but investigations have shown that paper contains numerous capillaries into which oil can penetrate but which do not pass entirely through the sheet, hence, would not be evidenced by a porosity test. Porosity is measured by passing a definite volume of air through a given area under a constant pressure. The Gurley densometer and the Gurley S.P.S. are instruments used in this measurement. The Bekk smoothness tester has an attachment for measuring this factor. Porosity as an indication of oil absorption or ink receptivity is not reliable when it is considered that coated papers that have very low air passage may have very good ink receptivity.

The Oil Absorption test is an attempt to measure the ink receptivity of a sheet and is fairly accurate for these inks which dry by absorption but does not necessarily indicate the ink receptivity of the surface. Various methods used to measure the absorption of a thin film of oil on the surface of the paper have been developed and are more reliable measurements of ink receptivity. However, since considerable pressure is applied on the press, hydraulic rules are in effect as well as capillary attraction. Probably the proof press is the best means by which the relationship between paper, ink, and press can be shown.

Moisture, another important characteristic should be closely checked because of the effect it can have on the sheet. Excessive moisture has been blamed for slow drying of inks and the resultant troubles. An excess or shortage of moisture tends to cause dimensional changes which in time causes many troubles in printing. Expansion and contraction of a sheet are very important factors. This is especially true in printing where multiple impressions are to be made and exact register is paramount. The moisture content of paper can be determined by oven drying and by several more rapid methods.

Opacity is the ability of the paper to prevent showthrough. This is especially important when both sides are to be printed. It is also necessary to prevent printing on the second sheet from showing through the first.

The different kinds of pulp have varying opacities. Groundwood has the highest and bleached sulphite has the lowest.

Probably the best way to control opacity is by adding mineral fillers. The opacity of a filler depends on its refractive index. Fillers commonly used are clays, talc, and calcium carbonate. Titanium dioxide is probably the best for opacity but the cost and supply prevents a heavy use of this material.

Of course, all the beneficial effects of the mineral filler is obtained at the same time.

The Bausch and Lomb Opacimeter using the contrast ratio method is probably the most common instrument used for measuring opacity.

Several other factors of paper should be mentioned as important to printability. Some of these are color, acidity or alkalinity, grit, ink coverage, and the mechanical perfection of the roll.

It has been said that the only way to test paper for printability is to print it. The various tests given above are actually tests for uniformity, but if the results obtained are correlated with papers that have given a satisfactory printed product, then certainly they should form a basis for specifications for printing paper.

The Influence of Pulp Characteristics in Printing Paper, by Bruce H. Wright, Crown Zellerbach Corp., West Linn

In the average run of printing papers no matter how much effort is put into improving the base sheet by coating or supercalendering, the quality of the pulp will be reflected in the finished print. If the pulp is of the wrong type or quality to begin with it is extremely difficult for the paper maker to make a good sheet of printing paper and for the printer to develop a high-quality print.

In setting up qualifications for the fiber components in printing paper, it is well to outline as follows the desired characteristics of the finished sheet which are directly affected by the pulp: 1. uniformity, 2. smoothness, 3. compressibility, 4. resiliency, 5. opacity, 6. over-all strength, 7. pick resistance, and 8. ink receptivity and absorption. These are felt to be the most important characteristics for the majority of printing grades manufactured today.

Of utmost importance is uniformity, since nonuniform pulp makes for nonuniform paper and if the printer does not have a uniform printing medium he is certain to have fluctuations in his print quality in spite of his adeptness in makeready and ink control.

In the opinion of many, smoothness is second only to uniformity in its effect on print quality. The contribution of pulp

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to smoothness comes in two ways, first from its effect on sheet formation and secondly from its ability to form a continuous surface. Long, highly fibrillated fibers tend toward wild formation on the machine. The resultant thick clots and thin voids provide an uneven surface which will not allow complete contact between the sheet and the printing plate. Although supercalendering may reduce this defect it cannot generally eliminate it and since the supercalendering actions hardens the high spots, they tend to refuse ink giving a motley appearance to the print.

Poor contact also results from the use of furnishes which do not have good continuity of surface due to the large dimentions of the fibers. It can be demonstrated with the fractions of any pulp that printability improves directly as fiber length and width decreases. Groundwood with its high percentage of short fibers and fines prints very well whereas softwood sulphite alone, with its preponderance of long fibers, prints comparatively poorly. In combinations the short fibers in the groundwood tend to fill in the gaps between the long sulphite fibers and the resultant furnish is fairly suitable for printing.

Compressibility, necessary for the cushioning effect in the presses to allow the printer some leeway in his makeready, may be obtained by using bulky pulps. However, no amount of cushion will help if the sheet is too rough.

Resiliency is needed for four-color printing so that the sheet will spring back to somewhere near its original caliper for the successful colors. Unfortunately pulps high in compressibility are generally low in resiliency.

High groundwood or unbleached chemical sheets usually have enough opacity to prevent showthrough of ink from the opposite side of the sheet. Any bleaching, refining, or supercalendering treatment will decrease opacity, however.

Pulp strength should be high enough for the paper to run on the machine and under strenuous conditions on high-speed presses. It must be capable of resisting the picking action of tacky inks. In order to fulfill these requirements some long strong fiber such as sulphite or kraft must be used even at the expense of printability.

Highly absorbent pulps tend to gray the prints and sometimes cause chalking. Ink absorption is reduced by closing the sheet and by supercalendering. However, excessive supercalendering hardens the high spots to such an extent that the sheet will be blackened and the surface nonreceptive, resulting in mottled printing.

It can be seen that pulp quality exerts considerable influence on the finished print and the manufacturer will do well to use his pulps to better advantage in obtaining good printing qualities rather than placing too much emphasis on subsequent operations.

Following the afternoon Technical Meeting there was a reception at the Crown Willamette Inn sponsored by the host mill. At the dinner, which was also held at the Inn, F. E. Church of the Production Department of *Time* and *Life* publications spoke on the editorial and production operations of these magazines. He said in part, *Time* and *Life* magazines are geared editorially to newspaper closing editorial deadlines and the production operation keeps pace.

Because *Time* is akin to a newspaper in magazine form, type is set and engravings made in three cities simultaneously, Philadelphia, Chicago, and Los Angeles, each Monday night. Subsequent printing and distribution of more than 1,500,000 copies from these cities is then effected for Thursday delivery throughout the country.

Life's text and engravings are processed in Chicago, and printing plate material shipped to Philadelphia and Los Angeles for printing and distribution from the three cities. Preceding this "current form" printing, 4-color covers and inserts printed in Chicago and Buffalo are shipped in advance for final assembly in the bindery. Final distribution of more than 5,500,000 copies of *Life* is effected for Friday arrival throughout the country.

Both operations are mechanized as much as possible: "Teletypesetter" is used for *Time* so that type is set in three cities simultaneously from one sending machine in New York; color presses print four colors on both sides of the paper web to deliver folded signatures ready for binding; binding machines are mechanically operated with trimmers, conveyor belts, and chutes for "assembly line" delivery to shipping platforms.

R. M. True

Empire State

Eastern District (November Meeting)

The November meeting of the Eastern District of the Empire Section was held at Glens Falls, N. Y., Nov. 16, 1950. The speaker of the evening was Harry S. Spencer of the Howard Smith Paper Mills, Ltd. at Beauharnois, Que. Robert Moynihan, Chairman, introduced the speaker who talked on the subject, "What We Do Not Know About Paper Manufacturing."

What We Do Not Know About Paper Manufacturing, by Harry S. Spencer, Howard Smith Paper Mills, Ltd.

"When your Mr. Crocker telephoned me a few weeks ago and accorded me the honor of being asked to speak to you, he gave me the choice of a subject. Half jokingly, I suggested—"What We Do Not Know About the Manufacture of Paper." He thought this had possibilities and I have proceeded accordingly. I was reminded of a book written by Oscar Levant entitled 'A Smattering of Ignorance,' and, in case we arrive at the point where we think that because we have spent the best part of a lifetime in the paper business we know a lot about it, it can be rather sobering to put down our thoughts on what we don't know. It can be a healthy sign if we have an acute awareness of our deficiencies, to perhaps spur us on to solve and overcome some of the fundamental problems of papermaking which still retains so much art instead of science. What we don't know causes poor quality, complaints, high costs and losses, or low profits.

"We must remember, however, that our customers are caught in the same squeeze as we are as they have had their labor costs increased, and the only way they can control costs is by speeding up their machines and this requires better and more uniform paper. When printing presses are speeded up as much as they have been, there are more exacting requirements of the paper with respect to dimensional stability, flatness, and other properties.

"We therefore find that we must provide a higher quality for our customers because they are speeding up and we also have to increase speed to keep out of the red ourselves.

"Those of you who have analyzed your raw material costs as a percentage of your total cost will know that, compared to prewar, the per cent of raw material assumes a higher percentage of the total than before. This means that the balance, which contains fixed charges and operating costs, gives us less to work on; so that the only opportunity we have to really reduce costs is through higher machine speeds and more economical use of labor.

"A bright young college graduate was once employed by one of the largest electrical manufacturing companies, devising process controls. One day he approached his boss and complained that the problem he was working on was still an art and not a science. His boss said, 'You get busy and change it from an art to a science or get to hell out of here.'

"Isn't that what most of us are trying to do? Whether it is our attempt to develop better equipment, speed up older machines, get better and more uniform control of stock, sizing, or physical tests—aren't we trying to convince our skilled workers that no matter how clever or experienced a man may be, he can be better with the proper type of controls, recording instruments and testing equipment; and, therefore, his best assurance of employment and promotion depends on his willingness to take advantage of these useful tools and so enhance his ability to make the most of his experience and skill.

"When I finished college I received a sound piece of advice: it was not to take a job that could be learned in a few months, but to take a job where one could spend a lifetime learning and still have something left unlearned. I think that we are all proud to have just such jobs in such a fascinating industry as is ours.

"The whole paper industry is founded on the principle of cellulose fibers suspended in a solution of water, and yet we don't know how to accurately indicate and record the consistency of a solution of stock.

"We start off with a raw material of wood which varies in density, moisture, and other properties. The fine paper industry, requiring rags, has to depend on a waste product

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habitually varying in strength, color, purity, and such. Consequently, one of our most serious problems is to take a raw material which we know varies over a wide range, and, by means of accurate treatment during processing and ultimate blending, end up with a finished product within specifications and sufficiently uniform to meet the customers' requirements, and this is a great demand to make of our personnel and our equipment.

"Because we don't know how to measure certain materials accurately, we buy pulpwood for the pulp mill, but we don't know how much—then we make it into pulp, but we don't know how much—then it is lapped and sold, or slushed into a beater, but we don't know how much—then it is dumped into a chest, but we don't know how much—chemicals are added, and we don't know how much is retained—then it is run over a paper machine.

"Those of us who try to reconcile the finished weight off the machine with the raw materials used, to figure the loss of fiber, realize how difficult it is. Then, because we don't know accurately what this is, we spend money on white water samplers and calculate the fiber and filler loss in the mill effluent. I know a mill which for months showed that they got more filler in the paper than they put in."

Statistics

"Statistical control is coming more and more to be used for analyzing problems. While most of us are not capable of understanding some of the higher mathematics involved, we all use statistics of some kind, even though they may be merely tests which require interpretation. Interpretations and conclusions can sometimes be quite wide of the mark.

"Some years ago a questionnaire was completed to cover information about 1000 men who were graduated from Yale and 1000 women graduates of Vassar. From these statistics it was found that 1.8 children had been born to the men of Yale and 1.6 to the girls from Vassar. One conclusion drawn was that men had more children than women.

"Then there is the story of the duck hunter who fired one shot just ahead of the duck and one shot just behind. On an average he killed the duck, but he had no duck to prove it.

"Let us examine the operations of a paper mill and see in more detail what we don't know. The scope of this paper does not include pulp-mill activity."

Stock Preparation

"(*a*) *Pulping, Shredding, or Breaking Up.* For years we have been content to break up pulp in the crudest manner: Only in the last few years has there been suitable equipment for disintegrating stock preparing it for beaters to reduce labor and prevent damaging expensive beaters by adding it in lap form, and there are many mills left which have not yet found a suitable method of stock preparation.

"I do not mean to approach this problem from an ultracritical or cynical point of view, because some wonderful progress has been made and is taking place right now, but rather—let us examine ourselves and understand how fruitful are the opportunities to reduce our shortcomings.

"Rag stock is still prepared in the same type of washing engine as used for many years, and I don't believe we have anything like the best answer to this problem.

"In the preparation of rag stock, the common washing engine appears rather a slow and expensive way of defibering rag from the textile down to the fiber. Cumbersome as it is, and inefficient as it certainly is as far as tearing apart is concerned, it nevertheless imparts certain qualities to the rag stock, and any change in design must take into consideration all of these factors. I know of a mill in England that prepared rag stock by means of serrated or saw tooth edges on their bedplates and roll bars. The rag stock was torn apart, or defibered perfectly, but when run on the paper machine it was so free that the qualities they needed in the paper had vanished. I know of several textile machines that will shred and disintegrate

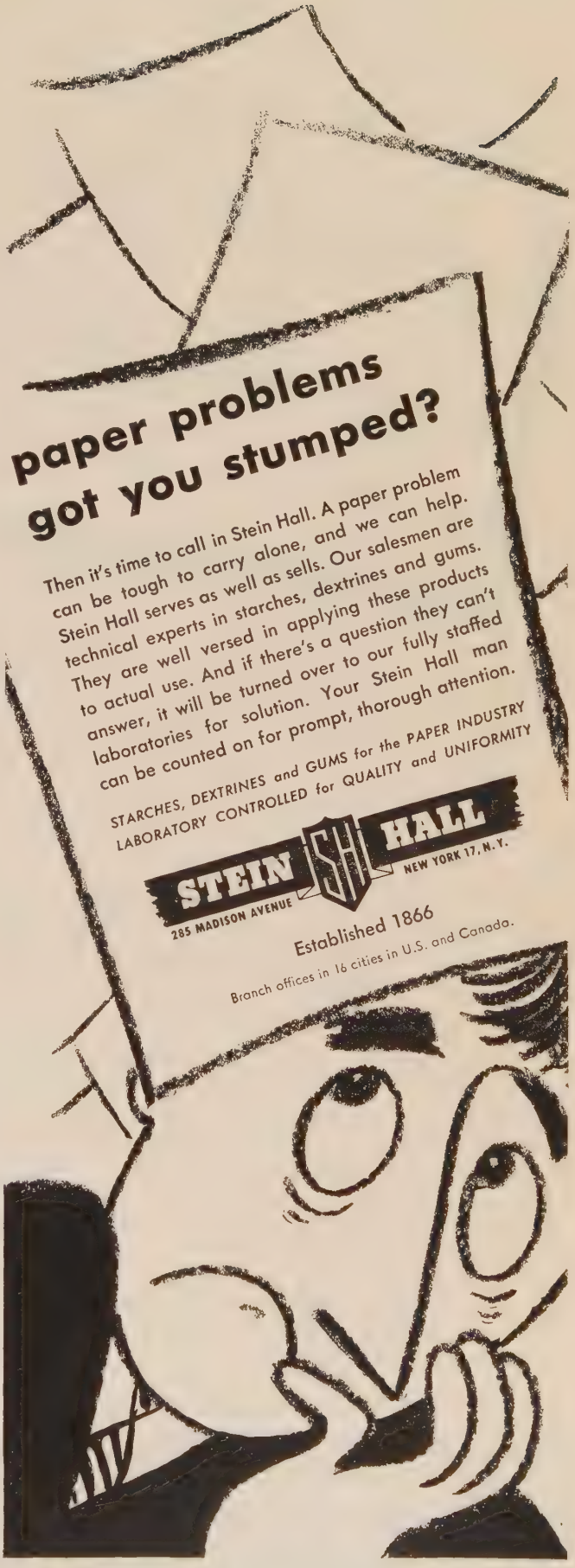
pieces of rag, but as yet we haven't found anything which will suitably supplant the rag washer. I think we will find that around many of these old processes, which we assume do certain things, there are valuable properties built into the stock, and new equipment must take all these things into consideration to make sure we produce similar quality in every respect.

"(*b*) *Bleaching.* Last Monday evening I listened to a lecture given by Dr. Purves, of McGill University, on Cellulose Chemistry. He said that theoretical knowledge of bleaching lags behind technical knowledge of what is known to work. I was surprised to hear him say that there was so much they do not know about bleaching. They don't know what lignin is. They don't know what the sulphite or kraft cook does to it. They don't know why two or three per cent of lignin is left in every cook. They don't know why multistage bleach is better than single stage, and why an acid bleach and an alkaline bleach are both necessary for best results.

"(*c*) *Beating.* Most of us can remember when the beating was done solely by the "feel" of the beater man and the superintendent, with tremendous variations of slowness and fiber length, in spite of some truly remarkable men who did amazing things in the way of control without any testing instruments and using only their own skill and experience. Beating "off the plate" required great care and the penalty for carelessness was expensive. Lack of knowledge of what actually went on in the beater spurred on the ingenious ones to devise tests which showed differences in stock, and the next step was to learn to control and gradually remove many of these variations. With this went improvements in design and production of equipment which was capable of being more accurately controlled. Many mills now test for slowness and fiber-length index to more accurately reproduce quality and maintain records for comparison. But how many times are the tests inaccurate, or how many times do we fail to get best results because we don't realize that supposedly identical beaters may, and often do, give widely varying results because of bedplates getting worn down. It is a batch process and old worn bedplates give far different stock from new sharp plates, and when the roll bars get too worn the circulation gets poorer. We don't always know the proper consistency to run or the most suitable peripheral roll speed.

"(*d*) *Addition of Chemicals, Fillers, and Dyestuffs.* There is a lot we don't know about this subject—the best order of chemicals and the best manipulation of color. The matching of color is probably as much of an art in papermaking as anything we do. We can't always agree on which of two sheets is brighter. There is a tendency for most customers to consider a bluish white brighter because of its bluish tinge, whereas a sheet with less blue may be brighter. Many people still confuse brightness with shade. Some will call a certain blue, bluer than another blue, which is quite meaningless. Before the war, when at the Wembley Exhibition in England, I bought a color-matching machine which was only about $150. The idea was to show the difference between a sample which would be paper or cloth, but it was not practical because the matching colors were glass and translucent, whereas paper reflected the light. Although it didn't do what we hoped, I believe there are instruments on the market which can do this, but they are very expensive and are certainly not developed to the point where they will replace human judgment by indicating accurately what dye and how much must be added or removed to make a match.

"Without criticizing other industries unduly, I think most of us realize there is a lot that the dyestuff industry does not know. We have never been able to get a completely satisfactory description of the dyestuffs we buy with respect to their development under beating, behavior under different pH conditions, changes when exposed to acid and alkali, tendency toward twosidedness, mottling, etc. Apparently the dyestuff manufacturers also have their problems turning their art into a science.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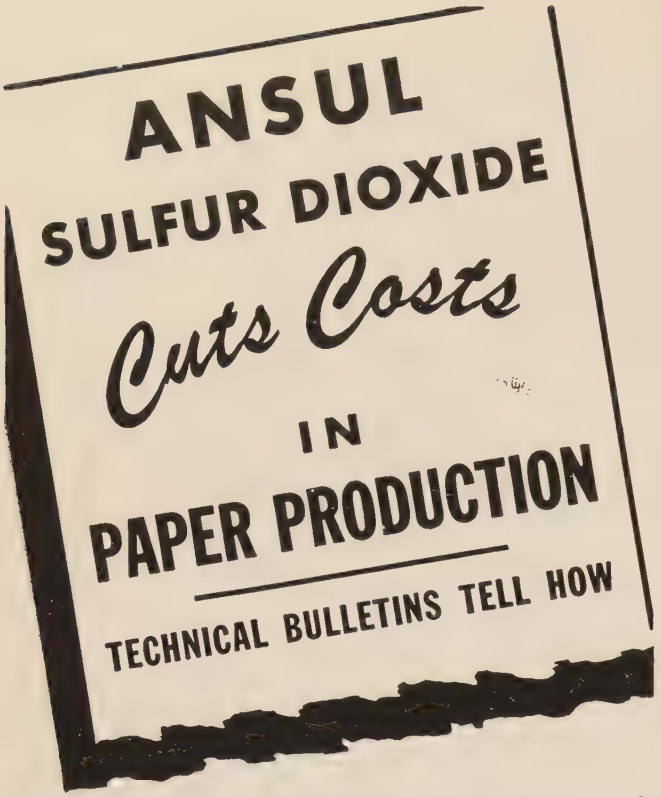

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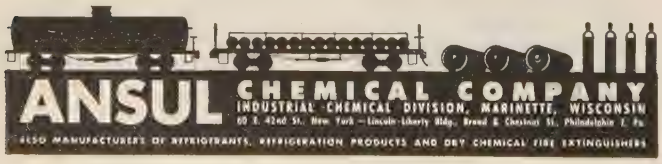
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ALSO MANUFACTURERS OF REFRIGERANTS, REFRIGERATION PRODUCTS AND DRY CHEMICAL FIRE EXTINGUISHERS

"(*e*) *Sizing.* I believe that every one of us here would be well qualified to make his contribution to a symposium of ignorance on the sizing of paper. I doubt if there is a more baffling or mysterious subject that is bound to confront us from time to time. Because we don't know how much size and alum to use, most of us use far more than is necessary just to play safe.

"(*f*) *Refining.* I'm pretty sure most of us here would readily admit that there is a lot we don't know about the relative merits of jordans, Claflins, Mordens, Sutherlands, and other refining equipment—to say nothing of the ramifications of different types of tackle.

"We don't know how to protect the filling from damage by foreign particles. We bought one device and designed one ourselves. They help, but I know of no mill that has not had trouble by losing a filling from a stray nail or a bolt.

"We don't know the best means of controlling the stock treatment. There are several devices to keep different variables constant. One of these sold, in my opinion, keeps constant something that should be allowed to change with varying stock conditions.

"We don't know how to maintain real accuracy in refining, and it is not necessarily a criticism of the equipment because it cannot do the impossible with changing stock. Most of us probably reverse the rotation of the plug weekly to remove the burrs and reduce the tendency to approach a cycle or batch process."

Pumping Capacity

"I don't know of anyone in the business of selling pumps or making paper who knows pumps so thoroughly that he can recommend a pump which will be trouble-free. We had trouble with plunger pumps causing lumps when the balls got out of round or the seats were worn too wide, and we've had trouble from centrifugal pumps getting air-locked, from the runner not having enough clearance, so that stock rolls in balls, takes excessive power and slows down the pump—causing weight variation or a burnt motor. With low stock in the chest, which means less head on the pump, a slightly lower head in the stuff box may result even with an overflow and cause weight variation."

Screening Capacity and Cleaning

"We don't know the best cut on screens to get cleanest paper—sometimes we sacrifice the finer cuts for production. We don't know how to get rid of stock lumps or rollers. We don't know whether we even need screens for all kinds of paper. There are mills running without any screens, and making good paper too.

When it comes to cleaning equipment, such as centrifugals and those involving a pressure drop, there is very much we don't know."

Headbox and Slice

"There are so many designs of each used today that one must conclude that there can be no one best for all conditions, but we cannot agree on how long the pond should be unless perhaps that it should be the shortest possible length. If we examine the many types of headboxes and slices, we must also conclude that there is a great deal we do not know about them.

"Papermakers don't agree as to whether the theoretical head is the proper head to run at, and, if not, how closely it should be adhered to. The supposedly ideal condition where the stock leaves the slice at the same speed as the wire (except for certain special papers) depends entirely on the judgment of the man looking at it, and it is quite easy to be fooled by surface water or wave motion.

"*Basis Weight Variation.* I wonder how many mills have come even close to real good operations in controlling basis weight. I don't think it's an extravagant statement to say that very few mills have even reasonably good control—I

mean being able to start a paper machine, whether it is at the beginning of the week or after a shutdown, within plus or minus 5% of specified basis weight (or very close to it), and then with one adjustment only get the correct weight and stay within plus or minus 2% all the time."

Wire Length

"These vary so much from mill to mill that we obviously do not know just how long a wire should be, even if we know the conditions under which it will operate. Most papermakers calculate their forming space as that area from the slice to the end of the shaking rail. Some, however, cover up 10 to 15% of the area with an apron and deprive themselves of the advantages of a longer wire, to say nothing of handicapping themselves with slices which do not give best results for even flow and good formation at higher speed.

"We don't know the proper slope for a wire. We have seen them as much as 30 inches downhill and several inches uphill. The tendency seems to be more and more to run them approximately level and adjust the head."

Shake

"This is surely something about which there is much we don't know. I have been in many discussions and heard experienced papermakers argue that a short fast stroke was required for a certain sheet, while others vigorously contended that a long slow stroke gave best results. Seldom, however, does the speed at which the machine is operated receive the consideration it deserves with respect to the shake. A TAPPI questionnaire has just been released on this problem and I hope that many of you will give your answers based on your experience.

"I was told recently that a company in England had a very old, shaky paper machine at one of their mills. When a traveler asked the reason for not modernizing it he was given to understand that they did not want to change it because they had told their customers that their paper could be made only on this particular machine."

Suction Boxes

"We don't know how the proper number of boxes for a paper machine, neither do we know the most suitable cover or the best type of slot opening, and some lively arguments have occurred regarding the best method of water removal. There is at least one method with an automatic feature, supposed to control something which I don't think should be kept constant at all."

Dandy Roll Operation

"Here is certainly one piece of equipment about which many of us lack thorough knowledge. I never met anyone who knew the best moisture content of the stock going under the dandy roll. Many skilled papermakers are excellent judges of appearances, but this remains an art instead of a science. I doubt if there are many who are aware of the extent of dandy-roll slippage and know how much per cent slower the dandy-roll surface speed is compared to the wire."

Suction Couch

"Here perhaps we have one of the most standardized pieces of equipment on a paper machine. Some mills are now running the wire around the roll, after and below the couch, and picking the sheet by suction off the wire where there is some slackness or cushion between these two rolls."

Presses

"It might be inaccurate to say we don't know too much about press rolls. Certainly many differences of opinion exist regarding composition of top rolls, and I doubt if optimum conditions of rubber hardness and crown exist in the majority

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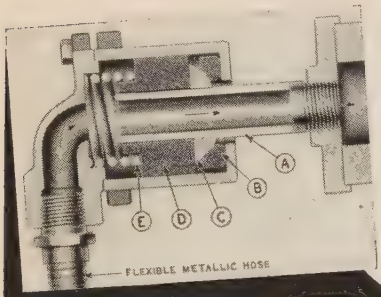

Nipple (A) is fastened to roll or drum. Carbon-graphite ring (B) effects seal against rotating collar (C). Guide (D) supports joint, is also of self-lubricating carbon-graphite. Spring (E) is for initial seating only; in operation joint is pressure-sealed.

Write for complete facts

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JOHNSON TYPE-S
Rotary Pressure **JOINTS**

The JOHNSON CORPORATION, 843 Wood Street, Three Rivers, Mich.

of mills. Those of you who have licked the problem of grainy edges probably have the most suitable plastometer reading and a crown which will leave enough moisture at the edges to prevent overdrying. There is no exact set of conditions which can be called perfect because the size and stiffness of rolls vary greatly."

Wet Felts

"I doubt if we know as much about our wet felts as we should. Most of us tend to rely too much on the manufacturers, who, in some cases, are reluctant to divulge specifications of weave. The result is that once in a while trouble occurs and the feltmaker discovers that someone has strayed away from standard practice, ending in grief for the papermaker. Many papermakers don't know how to tell which direction a felt should run by examining the nap, but most manufacturers now weave arrows along the edge to prevent this mistake."

Drying Section

"After the sheet is formed, it goes to various parts of the paper machine which are more standardized, although there are some differences of opinion about the size of driers, clearance between them, drier felts, etc. It is probably true that we don't know as much as we should about water removal and what goes on inside the driers and how often they should be cleaned to maintain good heat transfer. Those of us who have gone through the agony of properly eliminating condensate to prevent curling and cockling troubles realize that there are many things we don't know about relative humidity of air adjacent to top as compared to bottom drier felt."

Tub Sizing

"Some of the strangest things can happen to paper going through a size tub or size press that tell us we don't know all we should about this important part of a paper machine. We take regular tests on the soak or size pickup of certain specialty papers and find it difficult to do accurately, and still more difficult to interpret correctly the reasons for changing tests. The sizing may change because the moisture content of the paper entering the size press is altered, or because of slower or freer stock, formation changes, variation in the size itself, its temperature, acidity, press-roll pressure, etc. On one grade where resistance to moisture had to be measured with great accuracy, we found that the moisture resistance varied—in almost exact reverse to the pH under the wire.

"The driers after the size press have problems similar to those before, except keeping driers free of sizing scum from carry-over at higher speeds.

"The calender stack is quite standardized, although many differences of opinion exist as to whether the top roll should be crowned or just the bottom two rolls.

"The old-fashioned reels have been discarded in many places in favor of the Pope drum type so necessary for higher speeds.

"*Speed.* We don't know how much increased speed affects our burst, tear, tensile, fold, and other physical tests. We have all seen better-formed paper made at 300 feet than at 150 feet. We have seen paper watermarked at over 500 feet, which was better in every way than at lower speeds. Newsprint, kraft, and many grades are currently being run at tremendously increased speeds compared to a few years ago. Sometimes we have to sacrifice certain properties, but other times, by ingenuity and better-designed equipment, we can raise speeds and sometimes quality. It is such a highly complex problem that there can be no one stereotyped answer for it.

"*Costs.* Those of us who don't know our costs, don't know the weak spots where greatest energy must be applied. We must avoid the position of having the sales department stress a line on which we may be losing money.

Charles A. Dickhaut

Benjamin R. Newcomb

"In the endless urge for more production of higher quality to duce costs, various bottlenecks appear. If we have the ace, we can add to the drying capacity, but, instead of all iers, the addition of felt driers may save space. Then per-aps our beating capacity is insufficient and may have to be igmented by more beaters and something done by adding ore refining equipment. We then likely find that we have o do something about our screening and cleaning capacity.

"Then, have we the proper type of head design and slice to andle the increased number of gallons? When this is fixed, is ur wire long enough to get proper formation? Then how out our drives, transformers, boiler room, and so on?"

"What we don't know about the training of men is very vident whenever we have to make a shift in personnel nd the hunt is on for a man trained well enough to step up a better job which he can handle without additional long aining. Have we lost the ability to sell men the idea that ey should study for the job ahead before a vacancy occurs? o we put too much faith in our machines, or do we fail to alize that times are changing and that different methods of aining are a necessity?

"Aptitude tests, use of psychology for hiring and promotion, c., are attempts to do things better than we used to do. ven the most expert psychiatrists are the first ones to state ow much they don't know about its application to industry, ut this should be no discouragement when we know that so uch is being accomplished.

"The personal touch is still being needed more than ever, nd the better we can have our training methods, the better rganizations we can build and make our maximum contribu-on to society and industry."

astern District (December Meeting)

The December meeting was held the 14th, at Milfranks estaurant in Glens Falls. The evening was devoted to roblems of the paper coater and featured a discussion f coating equipment led by representatives of the John Valdon Corp., manufacturers of coating and allied ma-hinery. Robert Moynihan, Chairman, introduced the peakers who were Charles Dickhaut and Anthony Pomper. Ir. Dickhaut is in charge of the Research Division of John Valdron Corp. and is inventor of the Reverse Roll Coater, nd co-inventor of the Micro-jet and Synchroset Coaters, roducts of his company.

Mr. Dickhaut traced the development of coating machines tarting with the first brush coater in 1852 through knife oaters, roll coaters, reverse roll, air knife, and latest Syn-hroset coater. His talk was illustrated with slides showing chematic drawings of the various types of coaters together ith photographs of actual installations.

Mr. Pomper, Development Engineer, with John Waldron Corp., talked about recent developments in the field of poly-thylene extrusion apparatus for coating paper with this esin. He also described the operation of the Davidson-Pomper Pick Tester of which he was co-inventor.

Photo, Courtesy J. M. Huber Corporatio

Single Instrument Measures Smoothness, Porosity and Softness of Paper

You just interchange the lower test plates in the GURLEY-HILL S-P-S Tester to convert it into an instrument for testing either smoothness, porosity or softness of any type of paper, by measuring a given air-leakage over or through the paper.

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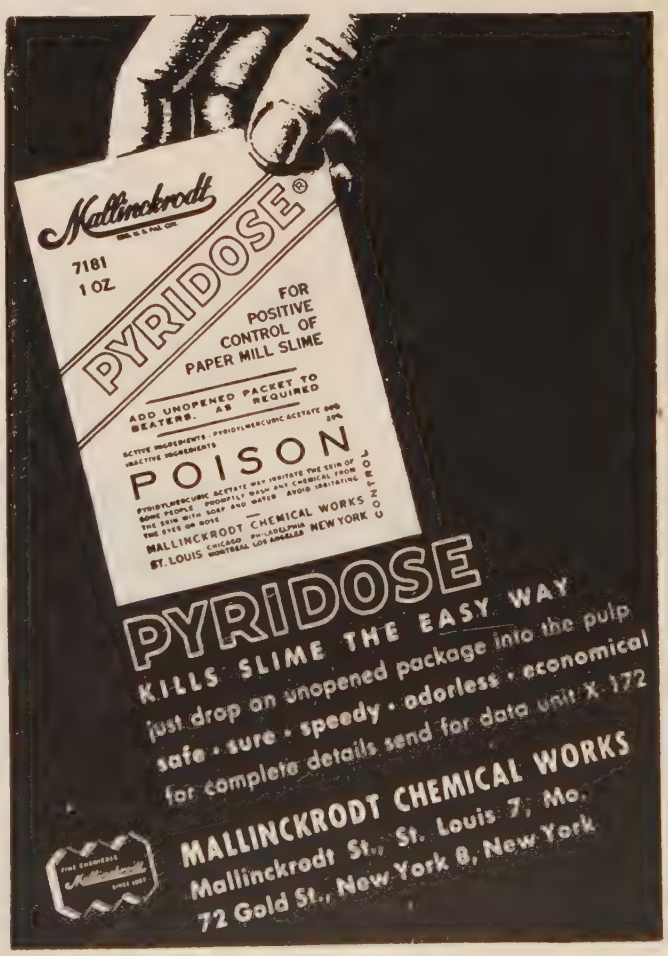
Bulletin No. 1400 describes the S-P-S Tester as well as other Gurley paper testing instruments. It's worth writing for today. W. & L. E. Gurley, Station Plaza & Fulton Sts., Troy, New York.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Benjamin R. Newcomb, President and General Manager of John Waldron Corp., was also present and joined the above speakers in answering the questions from the audience in the lively discussion period which followed.

The January 18 meeting was held at Milfranks Restaurant, Glens Falls, N. Y. This meeting was the Annual Ladies Night. The program featured "Adventures in Research," presented by a speaker from the Westinghouse Electric Corp., E. Pittsburgh, Pa.

ROBERT B. BLAKESLEE

Northern District (December Meeting)

The Northern District of the Empire State Section met as usual at the Woodruff Hotel in Watertown on December 14 for dinner and a meeting where representatives of the American Cyanamid Corporation discussed various developments in wax sizes for special uses. Harold Spector, of the St. Regis Paper Co., presided as Chairman. Ralph Prince, the Secretary-Treasurer of the Empire State Section, was present and gave a report of the finances and membership of the section.

Horace Spencer introduced the two speakers of the evening: George E. Fromm and Harold Sinclair who gave interesting talks and several demonstrations of the reactions of various wax emulsions on dilution and after application to various types of paper. Since their manuscripts had not yet been released for publication, no full summary or transcription of their papers can be given in this report. Among the problems discussed were shelf life and stability on dilution under various conditions of temperature, acidity, or alkalinity. Various procedures or suggestions for handling these types of emulsions were also mentioned. The samples distributed to those present included the following types and show the diversity of the applications:

Freezer wrap, with both beater and tub sizing
130-lb. fourdrinier sheet for milk bottle stock
24 point manila-lined board, with CMC on one side
Kraft interleafing sheet for steel sheeting piles
Patent-coated board using a combination of wax sizing with Zein
Nonsized sheet with wax emulsion used to eliminate chalky feeling from carbonate loading in sheet
Meat display board
Regular test liners

Northern District (January Meeting)

The Northern New York District of the Empire State Section met January 11 at the Hotel Woodruff for dinner and meeting. Harold Spector, Chairman, announced the appointment of Elmer Godfrey, Dexter Sulphite Co., as chairman of the nominating committee to suggest a slate of officers for the coming season. John Treadwell, Chase Mather, Horace Spencer, and R. Bodette were appointed to assist in this task.

Left to right: H. A. Spencer, Harold Spector, Harold Sinclair, George Fromm, and John Treadwell

Morris Meyers of the Socony-Vacuum Oil Co., the chairman of the planning committee for the annual salesmen's smoker to be held on February 8, announced plans for this meeting and the appointment of the salesmen who will assist him in the arrangements.

A. W. Infanger, Vice-President and Chief Engineer of the Cowles Co., discussed the Cowles Knifemill. Although this presentation was based on the paper presented at the New England Section meeting on Sept. 29, 1950, and recorded in the November issue of *Tappi*, interest was increased by the considerable amount of additional operational data from actual mill tests, together with numerous colored slides prepared from fibers treated with the special stains described by Francis L. Simons of the Crane Co. in an article on "A Stain for Use in the Microscopy of Beaten Fibers" (p. 312, Vol. 33, no. 7, July 1950 issue of *Tappi*). These slides indicated that the Cowles Knifemill had been able to accomplish the amount of refinement on rag fibers within 20 minutes that ordinarily resulted from about 36 hours in the old-fashioned beater. These slides with the fibers prepared with the two-color differential stain indicated the type and extent of physical changes in the fibers due to beating or mechanical refinement.

Dick Fang, the local Westinghouse Sales Engineer was instrumental in bringing S. E. Franklin, the Westinghouse consulting and application engineer of Syracuse to the meeting. Mr. Franklin described briefly the work of the Westinghouse High Voltage Laboratory at Pittsburgh and the part it plays in developing the safety features and increased service potentials in the electrical equipment used by industry, with special reference to the paper industry. Following this introduction a colored movie showed the facilities and several types of tests being made in this high voltage laboratory.

The speaker for the March 8 meeting will be Dr. J. T. Wilson, Chief Physicist at Allis-Chalmers Manufacturing Co., and the topic will be "The Engineer Can Help the Doctor to Save Your Life."

MISS L. V. HANS

Metropolitan District

The meeting of the Metropolitan District of the Empire

State Section was held under the chairmanship of Lewis S. Reid, Metropolitan Life Insurance Co., on Jan. 9, 1951, at the Fraunces Tavern in New York City, with the largest attendance of the season. Mr. Reid introduced Henry Perry, Editor of the *Paper Trade Journal*, and Chairman of the Empire State Section, who described the great devastation caused by the recent hurricane to the Adirondack Forests. He urged all listeners to write to their State Senators and Assemblymen asking them to pass the bill providing funds for salvage operations. Mr. Reid then turned the meeting over to Robert deS. Couch, General Foods Corp., who acted as moderator on the panel discussion on the general subject of "New Ideas in the Use of Paper for Automatic Packaging." It was emphasized that all speakers would talk informally for the benefit of the group and not for publication.

The first speaker of the panel was R. A. Morck, Chief Chemist of R. B. Davis Co. Dr. Morck outlined the procedure his company will follow in developing substitute packages for baking powder and Cocomalt, products of his company, if metal cans become unavailable. He also discussed the types of materials that will be considered and the relative costs of these materials in comparison with their present packages.

The next speaker was Niles H. Walker, Products Control Manager, Arnold Bakeries, Inc., who discussed the packaging of bread and rolls, outlining the protective requirements, the types of materials used, their strong and weak points, and the relative cost picture.

The third speaker was Walter Byrd, Chief of Package Development, Johnson & Johnson. He discussed packaging of dry yeast and dry diamalt, outlining the protective requirements of the products, the types of materials used to provide protection and the strong and weak points of packages now in use.

The final speaker was Mr. Couch who spoke on the packaging of coconut and dog food. His talk wound up the formal presentations and was followed by a question and answer period and discussion from the floor. The large attendance and the spirited discussion attested to the popularity of panel discussion meetings.

William Landes

Lake Erie

Over eighty papermakers and their wives were in attendance at LEPCA's first Christmas party held Friday, December 15, at the Hickory Grill in Cleveland. Excellent food served smörgasbörd style included juicy roast beef and lobster Newburg.

Frank Morrison of the Morrison Ink Company acted as master of ceremonies in presenting some excellent entertainment. Paul Ebert of the Ohio Boxboard Company, as song leader, developed many mediocre baritones and sopranos into concert artists soon after the good food had been settled by the initial warm-up song.

While all were in a festive mood a movie entitled "The Gift of Ts'ai Lun—Paper" was shown. All the male members were indebted to the Hammermill Paper Company for providing an opportunity to show their wives all about papermaking without trudging through a mill. The evenings entertainment was climaxed with some clever feats of legerdemain performed with the holiday spirit in mind, such as blank handkerchiefs spelling Merry Xmas with a wave of the hand. The evening was topped off by dancing in the club rooms till the small hours.

Paul Meissler, Chief Designer of the Hawkins Company, spoke at the January 19th meeting on the subject of "Package Engineering," followed by a short talk with the present world situation as its theme by Captain G. C. Briant, Commanding Officer of the Akron Naval Air Station.

The February 16 meeting will cover the subject of "Materials Handling" with Bill Hasselo, Assistant Manager, Container Corporation's Cleveland Plant, as program director.

Both meetings will be held at the Hickory Grill, 929 Chester Avenue in Cleveland. Cocktail and dinner hours are 6–7:15 p.m. with the meeting scheduled at 8:30 p.m.

R. L. Lewis

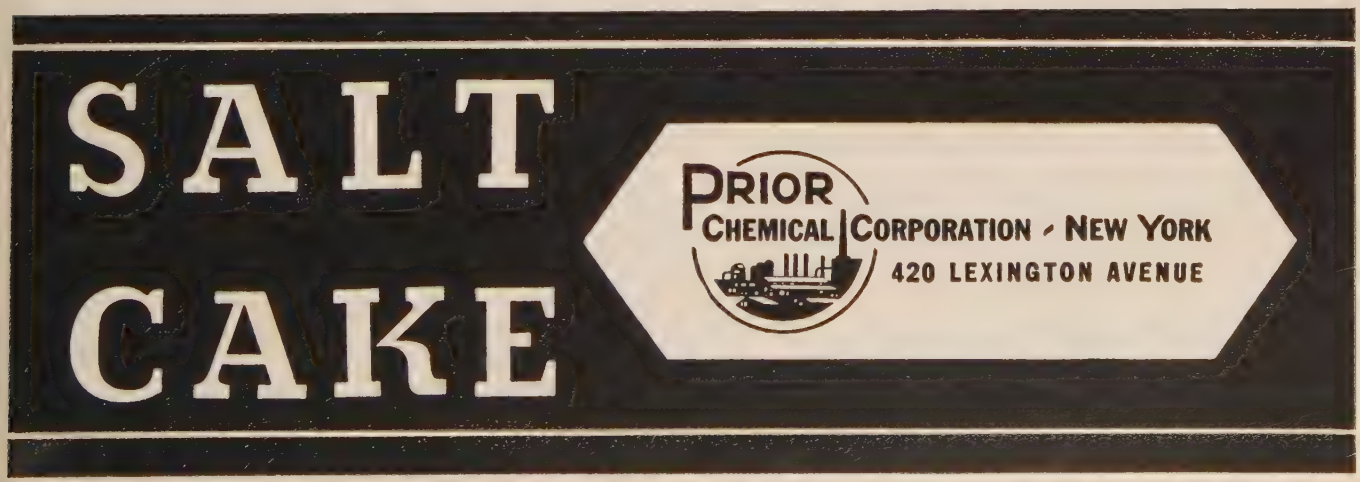
Ohio

The November meeting of the Ohio Section was held at the Manchester Hotel, Middletown, Ohio, on December 12th. The 125 members and their guests present were treated to an excellent lecture on "Fiber-to-Fiber Bonding" given by Herman F. Mark, Professor of Organic Chemistry, and Director of the Institute of Polymer Research, of the Polytechnic Institute of Brooklyn, N. Y.

Dr. Mark opened his address with the question: "Why and how can two fibers adhere to each other?" In explaining to the group the possible ways that fibers can bond together Dr. Mark cited four factors that enter into the adhesion of one fiber to another. These factors of adhesion are discussed in order below:

1. Mechanical

Mechanical attraction consists of the interaction of one fiber to another by means of the friction of their surfaces. Obviously interlocking or twisting of fibers can give some apparent adhesion. An example of this type of adhesion would be the long twisted fibers of rope which are held together by the friction of the fiber surfaces and the pressure of fiber against fiber because of the twisting the rope has undergone. In this case there is no actual attraction fiber to fiber but there is frictional resistance against separation. Other ex-



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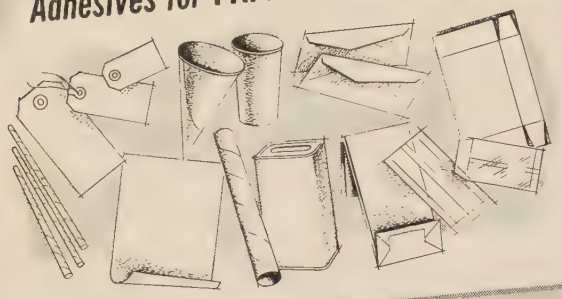

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amples employed by Dr. Mark to illustrate mechanical attraction consisted of glass and steel. When two pieces of glass or steel are pressed together there can be enough friction, depending on the pressure applied to the blocks, to prevent separation of the blocks parallel to their plane of union. Again in this case, there is no actual attraction; and the blocks may be separated by merely lifting one from the other.

2. *Polar Attraction*

When two fibers are pressed close together natural forces of polar attraction are caused due to the attraction of positively and negatively charged particles. Carboxyl and hydroxyl groups are responsible for polar attraction in cellulose. In this case we have actual forces of attraction in operation. Polar forces decrease rapidly with distance, and the distance cannot be greater than four or five Angstrom units for polar attraction to take place. In the case of polar attraction, a force of 2000 calories per mole is required to dissociate the attracting bonds.

3. *Hydrogen Bond*

When fibers are pressed still closer together, in the neighborhood of two to three Angstrom units, the hydrogen and oxygen atoms in adjoining cellulose units can attract and unite in close linkages of much stronger proportions. This form of linking is known as hydrogen bonding. In this case energies of 5000 calories must be expended to dissociate the bonds.

4. *Chemical Bond*

When cellulose fibers are brought together with distances as little as one and one-half Angstrom units, hydroxyl groups can be joined together with the elimination of water. Here a true chemical bond is formed requiring 30,000 calories for dissociation. In this case cellulose fibers can be interlinked by ether bonds. An example of this type of linkage is the parchmentizing of paper with sulphuric or phosphoric acids.

Herman F. Mark illustrating his lecture

In summarizing these four methods by which fibers can adhere one to another Dr. Mark presented a simple table listing the forces as shown below:

Type of bonding	*Order of distance*	*Calories per mole for dissociation*
Mechanical	Very large	Very small
Polar bond attraction	4–5 Angstrom units	2,000
Hydrogen bond	2–3 Angstrom units	5,000
Chemical bond	1–5 Angstrom units	30,000

It can be seen from the above that if the surface area decreases the bonds become stronger and stronger, and force required to break the bonds must be greater.

Having presented a brief theory of fiber-to-fiber bonding, Dr. Mark went on to discuss the importance of each bonding factor in the wet strength of paper. The discussion of these factors was in the order given above for the theory.

1. Mechanical adhesion fiber to fiber is not a molecular force of attraction, and any wet strength depends on the degree of intertwining and swelling of the fibers. If there is no swelling, strength is decreased by wetting. It is quite evident that the short fibers from which paper is composed cannot be twisted firmly together; and therefore mechanical bonding will not resist water.

2. Polar bonds are weakened by water, since water is a polar molecule and can enter into the polar bond itself. The polarity of water satisfies the hydroxyl and carboxyl bonds and thus eliminates the fiber-to-fiber attraction.

3. Hydrogen bonding is not effected as much by wetting with water because the distances involved are too close for the water molecules to come between the bonding groups. Hydrogen bonds are thus resistant to wetting; and this is the reason cellulose is not soluble in water. Thus cellulose cohesion is mainly due to the hydrogen bonding in the cellulose molecule. There are really very few hydrogen bonds uniting fibers in paper, while cellophane has many. Cellophane can be thought of as a mass of matted molecules and not fiber. The distances with which the cellulose units are separated in cellophane are thus very much less than the distances encountered in fiber-to-fiber bonding in paper. Cellophane swells in water but does not disintegrate; but an increase in the activity of the water by heating can cause cellophane to dissolve.

4. The ether linkage met with in chemical bonding is more resistant to water than is the hydrogen bond. As mentioned before, sulphuric acid or phosphoric acid will eliminate water from cellulose and establish ether bonds. These ether bonds give wet resistant papers, but also tend to create sheet brittleness.

In paper we have factors 1 and 2 and some of 3 contributing to fiber bonding. In most papers it is undesirable to have too many of No. 4 because of the resultant stiffness and brittleness of such a sheet. Hydrogen bonds are created mainly through fibrillation of the paper pulps during refining.

Other materials such as rubber and resins can be used with cellulose to give wet strength with the necessary stretch and elongation. To get this effect chemical groups are employed which give hydrogen bonding with the hydroxyl groups of cellulose. To gain flexibility it is desirable to use groups which have sizes in the order of 5 to 7 Angstrom units; since close distances such as one gets with ether linkages cause the strains which make for brittleness.

Groups giving good stretch and elongation consist of carbonyl and cyana groups both of which form hydrogen bonds with cellulose.

Dr. Mark illustrated the morphology of cellulose with some very interesting slides made from photomicrographs from the electron microscope. These slides illustrated the matted fibrils present in the primary cell wall of cellulose fibers from spruce, flax, and hemp. Slides were also shown illustrating the types of structure found in the secondary cell wall; it was pointed out that the primary wall must be ruptured in pulp refining if extrusion of the secondary wall fibrils is to give any fiber-to-fiber bonding. It was pointed out by Dr. Mark that fibril structure may be a most important feature of fiber-to-fiber bonding.

Following Dr. Mark's lecture the TAPPI men showed their lively interest in this subject of fiber-to-fiber bonding by the numerous questions asked of Dr. Mark. Some of these questions are answered briefly below:

Question: What range of distances is required for optical contact?

Answer: Optical contact is secured with distances in the order of one micron. The distances are thus rather large.

Question: Is there a basis for the attributed spiral structure of primary and secondary cell walls?

Answer: Yes, in some cases, as in cotton. Fibers from various species differ much in their structure, and also differ according to their age of development.

Question: Why don't fibers bond after they are suspended in acetone or other solvents?

Answer: Water is a small molecule, and on elimination of water the fiber-to-fiber distances are close enough to give hydrogen bonding. Acetone on the other hand is a larger molecule and can be removed from the fiber at a much lower temperature. Fibers soaked in acetone are farther apart, and when the acetone is removed no hydrogen bonding is formed.

Question: Can the ether linkages be proved to exist in parchmentized paper?

Answer: Yes, because these papers will not dissolve in normal cellulose solvents.

Question: Would carboxyl methyl cellulose give increased bonding?

Answer: Yes, carboxyl methyl cellulose gives carboxyl groups which are apart from the chain. They can thus bond and nest with cellulose.

Question: Has the thio linkage been used in cellulose?

Answer: The cellulose xanthate group can be insolubilized if oxidized.

Question: Would the thio bond be rigid or flexible?

Answer: Yes, the thio bond would be a desirable flexible bond except for the fact that it is not too stable to heat.

Question: If acetone cannot penetrate sufficiently is it a matter of dielectrical strength?

Answer: Yes.

Question: In wet-strength papers are the bonds of an ether linkage type?

Answer: We know that alkaline washing of wet-strength papers loosens the bonds; so they are not all real chemical linkages.

Question: Is the easy bonding of purified hardwood semichemical pulp due to the increased number of hydroxyl groups present in the hemicellulose groups?

Answer: Yes, in general this probably is true.

Question: How thick is the primary cell wall?

Answer: About $^1/_{10}$ of a micron.

Question: What is the chemical construction of the primary cell wall?

Answer: This matter of chemical construction has not been fully solved but is regarded to be cellulose with hemicellulose deposited both outside and inside the cell wall and with lignin deposited also on the outside.

Slime Control in Paper Mills—Some Questions and Answers

THERE have been many papers and discussions on the subject of slime control, but for most mill operators such information is widely scattered and difficult to find. The following makes it easy to find an answer to questions that most frequently occur.

QUESTION: What is slime?

ANSWER: Slime is a gelatinous accumulation which may be caused by microorganisms in which case it is referred to as microbiological slime. However, gelatinous or slimy accumulations can also be formed by highly hydrated fibers and chemicals used in the furnish. It is often difficult to distinguish the difference by visual observation in the mill.

QUESTION: What are microorganisms?

ANSWER: Microorganisms are living things which, because of their size, require a microscope for observation and study.

QUESTION: What is meant by microbiology?

ANSWER: It is the study of the size, development, and distribution of microorganisms and work which they do (putrefaction, decompositions, slime). Microbiological problems then pertain to such growths and activities.

QUESTION: How can the causes of slime be determined?

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ANSWER: They can be determined only by microbiological and chemical investigation of the slime deposits formed. Analysis of deposits shows their actual compositions while analysis of stream or slurry samples indicate sources of potential deposits.

QUESTION: Are chemical and microbiological slimes related?

ANSWER: Yes. Chemical slimes or deposits harbor microorganisms which increase the severity of both chemical and microbiological slimes. Growth of microorganisms frequently acts as a binder to hold together and accumulate chemicals and materials of furnish.

QUESTION: How do microorganisms cause slime?

ANSWER: Slimes of microbiological origin are the viscous and gummy growths of microorganisms, composed of mucous secretions of the organisms in addition to the organisms themselves.

QUESTION: Are all bacteria slime forming?

ANSWER: No. Outside of the cell wall of most bacteria there appears a capsule, slime layer, or sheath which varies in thickness, the consistency and chemical composition of which depends on the species of bacteria, growth conditions, and conditions in the mill. Some capsules never reach the stage where they contribute appreciably to deposit accumulations.

QUESTION: What is the general control classification of microorganisms?

ANSWER: Nonspore-forming bacteria, spore-forming bacteria, yeast-type fungi, and thread-type fungi (molds).

QUESTION: Why is microorganism control in paper mills more difficult than in other industries where sanitation is practiced?

ANSWER: Conditions such as source of food, moisture, shelter, and materials conducive to slime formation are more readily available to microorganisms in paper mills. Microorganisms which are usually not considered slime formers in other industries frequently produce slime growths in paper mills. This increases the varieties and numbers of microorganisms which require control. Also the extremely heavy slime masses formed retard the ability of chemicals to penetrate into the slime and kill the microorganism.

QUESTION: How do nonspore-forming bacteria form slime?

ANSWER: The capsules formed around the body of the organism stick to mill surfaces and to each other to form voluminous slime growth.

QUESTION: How do nonspore bacteria multiply?

ANSWER: Nonspore-forming bacteria multiply by binary fission (splitting) so that one bacterium forms two, two form four, etc.

QUESTION: Are nonspore-forming easily killed?

ANSWER: Nonspore-forming bacteria are usually easily susceptible to killing by many chemicals and temperatures above 140°F. However chemicals must be able to penetrate the protective capsule. Nonspore-forming bacteria are killed by the heat of the drier rolls during the drying operation.

QUESTION: What other troubles may nonspore-forming bacteria cause?

ANSWER: They also cause odors in paper, decomposition of stock and sometimes corrosion of metal surfaces.

QUESTION: How do spore-forming bacteria multiply?

ANSWER: They also multiply by splitting. While the bacterial spores germinate like seeds, spore formation in bacteria is not primarily a method of reproduction; its chief function is to enable the organism to withstand unfavorable conditions.

QUESTION: How do spore-forming bacteria form slime?

ANSWER: They form slime in two ways: (1) by their surrounding gelatinous capsule or envelope and (2) by entangling other bacteria and materials within their stringy structure. One of the most troublesome types is sometimes called "hay bacillus."

QUESTION: Are spore-forming bacteria susceptible to chemical treatments?

ANSWER: They are highly resistant to heat and chemical treatments. The spore or seed is undoubtedly one of the best heat insulating materials known. The actively growing well-hydrated stage of bacterial growth is usually susceptible to treatment.

QUESTION: How can spore-forming bacteria be controlled?

ANSWER: By using treatments which are specific for these groups and are chemically stable so that the treatment is present and active during their growing stage. These chemicals must also have the ability to penetrate the slime masses and to reach the living organisms embedded in the slime masses.

QUESTION: What other effects do spore-forming bacteria have on papermaking?

ANSWER: Their presence results in higher bacterial counts in finished paper products because they are not killed on the driers. Some types also cause odors.

QUESTION: How do mold-type fungi grow?

ANSWER: They are plantlike growths with branches and spore heads containing spores or seeds which form new growths.

QUESTION: How do they form slime?

ANSWER: Because of their large filamentous or branchlike characteristics they become serious slime formers by entangling and trapping fiber, other microorganisms, and materials of the furnish.

QUESTION: Are fungi resistant to treatment?

ANSWER: They are resistant to some chemicals but chlorinated phenols can control them if the chemical is able to reach the microorganism. It thus becomes necessary to use some method of dispersing or penetrating the slime accumulation to reach the microorganism. Practical sanitation and "good housekeeping" are very important in their control.

QUESTION: Can fungi growths cause difficulties other than slime?

ANSWER: Yes. Under conditions favorable for their growth they can decay, discolor, and produce odor in pulp and paper.

QUESTION: How do yeast-type fungi reproduce?

ANSWER: They usually reproduce vegetatively by budding as illustrated. A bud or buds will grow from the main stock or branches of the organism and when these buds reach maturity they break off and produce additional growths.

QUESTION: How do yeasts form slime?

ANSWER: Usually by adhering together and on walls of equipment.

QUESTION: How can yeast-type fungi be controlled?

ANSWER: By a good sanitation program including thorough and regular mechanical cleaning of the system, particularly spots which may be stagnant, and regular application of chemicals for control.

QUESTION: What is the relative size of various microorganisms encountered?

ANSWER: The unit of measurement used in microbiology is the micron which is 1/1000 of a millimeter or approximately 1/25,000 inch. The average bacterium is commonly 1 to 5 mu in length. A man magnified to the same degree necessary to study bacteria would be 1 mile high and 50 yards wide. Molds and yeasts, which vary greatly in size, are much larger. A mold-type fungus may, because of its size, contribute more to slime formation than several thousand bacteria.

QUESTION: Do microorganisms acclimate themselves to chemicals?

ANSWER: Yes. However, true adaptation or acclimatization seldom occurs in a paper mill system. The increased resistance of microorganisms in a mill system is usually the result of a process of selection whereby the sensitive organisms are eliminated and the inherently resistant organisms survive and reproduce, thus building a population which is resistant to the original treatment regime.

QUESTION: What variables affect slime growths?

ANSWER: Growth varies with the seasons, rainfall, stream pollution, pulp and furnish changes, and careless application of chemical treatment and sanitation control.

QUESTION: How can these variables be controlled?

ANSWER: By diligent attention to a slime control program which is sufficiently flexible to quickly neutralize changes which may occur.

QUESTION: Why, when a slime-control program has been effective, are there sudden outbreaks of slime even though chemical and cleaning programs have not been changed?

ANSWER: There is competition for food among various organisms in a system and if one type is killed off another type which is not susceptible to the slime-control program in force may flourish and cause slime, usually of a different appearance.

QUESTION: How can this be controlled?

ANSWER: By using a combination of chemicals that controls all types which may be encountered. Frequently chemicals have a specificity for certain groups of microorganisms. For general control, combinations of specific treatments are more economical than high concentrations of nonspecific chemicals.

QUESTION: What is one of the most important factors in maintaining good slime control?

ANSWER: General mill cleanliness or "good housekeeping" is a "must" in good slime control. There is no substitute for "good housekeeping." Successful slime-control programs are those in which chemical applications are used as an aid to "good housekeeping."

QUESTION: Is a program of cleaning and chemical treatment applied during week-end washups sufficient to prevent slime trouble?

ANSWER: While thorough wash-up programs are basically important they cannot in themselves be depended on to prevent slime. Rapidly growing organisms frequently cause trouble unless chemical treatments are consistently applied. The presence of inaccessible places and dead spots in a mill system further emphasizes the importance of consistent chemical application.

QUESTION: Is there an equation for a good slime-control program?

ANSWER: Yes. Complete control requires a total of so much sanitation control, human effort, and slime treatment. This can be expressed as:

$$\text{Slime control} = \text{sanitation control} + \text{human effort} + \text{slime treatment}$$

If any of the three on the right-hand side of the equation are reduced, the other two must be increased or the efficiency of the slime-control program will be lessened.

QUESTION: Does foam aggravate slime growth?

ANSWER: Indications are that foam may stimulate slime growth and aggravate slime conditions. It has been observed that foam deposits on the sides of service chests or other equipment provide a favorable basis for activation of growth on the part of the rapidly growing bacteria. Where foam deposits are neglected over appreciable periods, a variety of microorganisms finds opportunity for growth.

QUESTION: In planning a slime-control program what should be considered?

ANSWER: The types of troublesome organisms, focal points of infection, physical characteristics of the mill, and means of supervision to adjust treatments for changes in the types of microorganisms present in the mill system.

QUESTION: How can troublesome organisms be determined?

ANSWER: Only by a trained bacteriologist. Samples are taken from proper points in a mill system, and the bacteriologist can analyze and classify organisms and report on the relative ability of the various classes of organisms to form the slime growths.

QUESTION: Is a microbiological analysis accurate?

ANSWER: It is fairly accurate. It is not as precise as, for example, chemical analysis of water or scale but it usually is not necessary to account accurately for all organisms present. The relative amount and the kind of most prevalent organisms are the essential information.

QUESTION: What significance does a microbiological examination have?

ANSWER: A microbiological examination (1) points out the most active slime-forming organisms so that correct treatment can be chosen and (2) indicates places and sources of contamination or incubation in the system so that treatment can be correctly applied to discourage and prevent growth. Control by prevention is far more efficient than attempting to kill the organisms after their multiplication and growth have progressed actively in the mill system.

QUESTION: Do total counts have any significance?

ANSWER: They indicate potential ability of the systems to support microbiological growth but do not necessarily tell whether slime trouble will be experienced. Total counts may be helpful in pointing out places where microbiological growth and decompositions do take place.

QUESTION: In order to develop a satisfactory control program, is it necessary to make the microbiological analyses at the mill?

ANSWER: Not necessarily. Counts will change in transit but a great deal of investigational work has shown that samples in transit for 3 days, which is an average expected period, seldom show a change of 5 times greater or less than the original count. Generally accepted procedure is to consider only a full logarithmic unit (10 times) as a significant change. Under any condition it is impossible to count all organisms present. In general then it can be said that even if changes occur in microorganism counts in transit, results do not lose their

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value because they still indicate types and activity of microorganisms.

QUESTION: What other information is advisable in order to plan a slime-control program?

ANSWER: An engineering survey comprising a flow diagram, data on raw materials, fiber and chemical furnish used, water tests, water consumption rates, points and quantities of white water sewerage, system temperatures, pH, and a general description of operation of the mill.

QUESTION: Why is an engineering survey necessary?

ANSWER: It is useful in helping select type of treatment, method of application, point of application, and places in system which require close supervision to prevent trouble.

QUESTION: Are both microbiological and engineering surveys necessary?

ANSWER: Yes. The microbiological survey is functional and indicates what chemicals should be used while the engineering survey is operational and indicates where the chemical should be applied. If one is neglected, the value of the other is greatly reduced.

QUESTION: What in general can be said about selection of chemicals for control of microorganisms?

ANSWER: Killing or inhibiting chemicals should be able to control widely varying types of organisms; they should be long lasting and chemically stable; they should be easy to apply and not be harmful or irritating to humans in quantities used in a mill system.

QUESTION: Do all materials used in a slime-control program have to be germicidal to microorganisms?

ANSWER: No. Sometimes a chemical by itself has little if any germicidal effect on organisms but it greatly increases the activity and effectiveness of the active germicide. This phenomena is called "synergism" and is the cooperative action of two materials so that the total effect is greater than the sum of the two effects taken separately. In addition any chemical which will disperse or penetrate slime masses improves the effectiveness of the active germicide.

QUESTION: What other actions are helpful in increasing the efficiency of microbiological toxicants?

ANSWER: Dispersive and penetrating actions are necessary so that the toxic chemicals are able to reach the cell of the microorganism. Any chemical which increases the activity of the toxic chemical increases its efficiency. In addition, any chemical which disperses or scatters the toxic chemical in a system increases its killing efficiency because a greater number of microorganisms are contacted and killed.

QUESTION: Is a laboratory evaluation always indicative of the true efficacy of a slime-control formula?

ANSWER: No, because microorganisms seldom exhibit the same characteristics in a laboratory that they do in their natural environment, i.e., a mill system. Laboratory evaluation nearly always provides ready contact of the germicide with the cell and does not accurately evaluate the ability of a material to penetrate the slime mass to reach the living cell.

QUESTION: Of what value then, is a laboratory evaluation of slime treatments?

ANSWER: It does give a relative comparison against some other disinfectant and tends to indicate relative specificity of chemicals on various classes of microorganisms.

QUESTION: What are some of the more popularly used slime-control chemicals and what general purposes do they serve?

ANSWER: Chlorine is used extensively in fresh water and white water. Its advantages are rapid action and low cost while its main disadvantage is that chlorine is used up rapidly in the presence of the organic materials common in a mill system.

Copper sulphate has been used as a fungicide but is corrosive and difficult to maintain in an effective form throughout the system.

Phenolic derivatives such as tri, tetra, and pentachlorphenate or chlorophenolphenate have a specific toxicity toward certain types of organisms. They are exceptionally good for spore bacteria and fungi at a low cost. They are not corrosive. They require longer contact time with the microorganism than some of the other chemicals.

Mercurials of the organic type are useful because the organic mercury is stable and therefore more available for reaction. Commonly used are chloromercuric phenol, phenyl mercuric acetate, and others of a similar nature. Advantages are their rapid reaction and specific effectiveness on some types of bacteria, particularly nonspore bacteria. Disadvantages are their high specificity for only certain organisms and in low dosages they can act as a stimulant to growth of microorganisms. Certain precautions are advisable for handling and using mercurials.

QUESTION: How can slime control be evaluated in a mill?

ANSWER: By visual observation of conditions throughout the mill, by microbiological counts in mill streams or by slime boards which have been used for many years in one form or other.

QUESTION: Do bacterial slimes which form films on slime boards affect the formation of fungus slimes on these boards?

ANSWER: Yes. Frequently slime boards tend to develop films of bacterial growth which resist and often inhibit fungus growths. Many mill operators report that invariably slime boards become coated with gelatinous growth of bacteria which prevents fungi from developing characteristically.

QUESTION: What can be done to obtain more accurate information on fungus slimes when bacterial formation is present?

ANSWER: It is possible in a mill system harboring fungi to locate boards in places where development of bacteriological films is sufficiently delayed to enable fungus growths to attach and form slimes.

QUESTION: What form of treatment should a slime board receive to make it most useful?

ANSWER: The slime board should receive the same chemical treatment as the rest of the system. If a less effective treatment or a more drastic application is used, the results obtained are frequently without significance. Instances occur where the unit builds up abundant growth in a system relatively free from slime-forming organisms. The converse has also been observed. In the former case the units were cleaned but not subjected to the killing concentration of chemical used in the mill system. In the latter instance the units received a sterilizing treatment far more drastic than would be economically possible in a mill system as a whole.

QUESTION: When slime-forming types have been reduced to one persistent growth which resists all common control treatments tried, what should be the next step?

ANSWER: First call upon a microbiological laboratory to determine the identity, significance, and actual resistance of the organism. Second, locate its breeding places and expose these spots to thorough cleaning in conjunction with a suitable chemical agent selected because of its specific action on the troublesome type of organism.

QUESTION: Will slime-control treatments also prevent decomposition of fiber in storage chests which may be reducing pulp yield?

ANSWER: Properly chosen and controlled applications of slime-control treatments will effectively prevent decomposition of fiber in storage chest. Halfway measures will control neither slime nor fiber decomposition. It is important to select the right treatment and make certain the pulp mass is uniformly treated and preserved.

QUESTION: Will rot from wood cause trouble later in the mill system when these logs are pulped?

ANSWER: The typical wood rots do not as a rule form slime. They will, however, reduce pulp yields. However, numerous types of bacteria and commonly occurring molds growing on these logs sometimes withstand the heat of grinding and produce slime growths in the mill. Control of this phase should

ve a definite place in the program followed by mills manu-
:turing or utilizing groundwood.

QUESTION: What are other sources of microbiological in-
:tion?

ANSWER: Most organisms are waterborne but they may
:o come into the mill through air, pulp, and other materials
the furnish.

QUESTION: Do microorganisms contribute to corrosion in a
ll system?

ANSWER: Yes. They may contribute to corrosion in one
more of the following ways: (1) directly influencing the
eed of the reaction which takes place when a metal corrodes;
) removing the natural protective film on the surface of the
etal; (3) creating a corrosive condition through the prod-
ts they form in living. For example, sulphate reducing bac-
ria can reduce the sulphates in water to an acid hydrogen
lphide which is very corrosive; and (4) establishing a phys-
l barrier on the metal which sets up a battery action (con-
ntration cell) which leads to electrolytic corrosion of the
etal.

QUESTION: How can microbiological corrosion be con-
olled?

ANSWER: It can only be controlled by strict adherence to
ean-up programs and applications of slime treatments.
rculation of hot solutions of detergents and slime-control
emicals during washup is extremely helpful in ridding the
stem of these deposits. This is particularly true of con-
mination in inaccessible areas.

QUESTION: What causes musty or sour odors in paper?

ANSWER: This type of odor is caused principally by bac-
rial action in paper or stagnant furnish. Molds and prod-
:ts of molds also occasionally cause "musty or moldy" odors.

QUESTION: Does odor develop in paper in storage even
ough there is no odor immediately after paper is made?

ANSWER: Yes. Paper can be contaminated in storage and
n develop an odor. Frequently odors develop and become
ore intense after some time due to slow progressive decom-
osition in the stored rolls.

QUESTION: How can the paper mill prevent odors from
is source?

ANSWER: (1) Check stagnant areas in a mill system; (2)
eck pulp furnish and reject smelly pulp on quality orders;
3) clean system more frequently and thoroughly; and (4)
nprove slime-control program in mill.

QUESTION: How can chemical odors be prevented?

ANSWER: (1) Reject bales of paper with off odors in qual-
y orders; (2) open the white-water system occasionally to
ilute contamination; (3) wash pulp as completely as pos-
ble; and (4) use a small amount of alkali in pulp prepara-
on which helps remove phenolic inks which are major of-
enders in waste paper pulp.

QUESTION: Do phenolic-type slime treatments contribute
o odor?

ANSWER: Not when properly manufactured, selected,
sed, and applied for slime control. Some phenolic deriva-
ves have no objectionable odor at all. Even the most odor-
us materials do not contribute to odor in paper unless very
igh localized concentrations are experienced in the paper sys-
em because of improper application. Any properly planned
ime-control program should include this consideration, and
o difficulty will be experienced from this source. The use of
ispersing agents helps prevent high localized concentrations.

QUESTION: What effect does a slime-control program have
n bacterial counts in paper?

ANSWER: A properly planned and managed slime-control
rogram assists in reducing bacteria counts in paper in the
llowing way: (1) controls and rids the system of heat re-
sting bacteria which are not killed by drying; (2) elimi-
ates slime accumulations in the system which harbor and pro-
ote abundant growth of bacteria; and (3) aids in efficient
dherence to the mill sanitation program.

QUESTION: What is "moldproofing" of paper?

ANSWER: It is the incorporation of a suitable antiseptic into the paper in an amount sufficient to prevent the growth of microorganisms on the paper whenever the paper is exposed to microorganisms.

QUESTION: Are slime-control programs and "moldproofing" programs related?

ANSWER: They are not necessarily related. Slime control is directed at inhibition and control of microorganisms in the wet slurry phase of papermaking, whereas moldproofing applications are directed to the manufactured paper itself.

QUESTION: Will slime-control programs have any mold-proofing effect in the paper?

ANSWER: Because efficient slime-control programs help eliminate resistant microorganisms they do prevent contamination of the paper from the mill system so the paper is not subject to deterioration due to contamination from this source. It will not be protected from subsequent contamination in storage or handling.

QUESTION: How can mold-resistant paper be evaluated?

ANSWER: It can be evaluated in the laboratory by subjecting the paper to mold spores or "seeds" and providing ideal conditions such as food source, moisture, and temperature, for accelerated growth of the mold. If no growth occurs under these conditions, the paper is then considered mold resistant. Another method is to test it under simulated field conditions.

QUESTION: Which method is preferable, the field test method or the laboratory evaluation?

ANSWER: The field test method provides more accurate information but is too slow for practical purposes. It may take a year or more to complete the test. Laboratory evaluations on the other hand provide relative data which become available quickly, and experience has shown that it is satisfactory.

QUESTION: Are there different degrees of mold resistance?

ANSWER: Yes. Mold resistance of paper like water resistance of paper is a relative factor and depends on the chemical used, the quantity of chemical used and the method used to apply the chemical.

QUESTION: What are some methods of applying chemicals for mold resistance?

ANSWER: Two methods have been commonly used: (1) application in the wet end of the paper manufacture, similar to engine sizing, and subsequent precipitation of chemical to the fiber; and (2) surface application in size tub or on calender stacks.

QUESTION: What are the advantages of each?

ANSWER: Precipitation in the fiber at the wet end affords better and more uniform protection through the entire paper while surface application only protects the surface. The surface method of application is less expensive.

QUESTION: When making lined board can complete protection be obtained by applying protective chemical to the liner only?

ANSWER: No. Just as a chain is no stronger than its weakest link, so is mold resistance of a board no stronger than its weakest component. Molds have ability to reach down, through protected layers if necessary, to obtain nourishment and conditions for growth.

QUESTION: Have slime-control programs proved themselves useful to paper mill operation?

ANSWER: Yes. In the last decade slime control has become a part of the practical business of papermaking. Modern technological advance has shown that slime problems tie in closely with mill operation and production efficiency. Slime control is intimately associated with mill problems of pulp yields, sizing, sheet formation, felt life, foam, pitch, corrosion, odor control, and recovery systems. Thus, a modern control program accomplishes far more than slime prevention. It is an accepted procedure for efficient production of pulp and paper under uniformly favorable operating conditions.

HARVEY E. BERG AND J. RAYMOND SANBORN
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Contrast Gloss of Paper at 57.5°

Revision of TAPPI Official Standard T 424 m-45

(THIS STANDARD IS UNDER THE JURISDICTION OF THE OPTICAL PROPERTIES COMMITTEE)

Gloss is the geometrically selective reflectance of a surface responsible for its shiny or lustrous appearance. (Surface reflectance is commonly at a maximum in or near the geometric directions in which a mirror would reflect light.) The following contrast-gloss method, based on the Ingersoll Glarimeter, is intended primarily for white and nearly white papers, such as newsprint, writing, and ledger, having a low gloss. It is recommended for measurements of the tendency of these papers to glare when observed under improperly distributed light. It is not recommended for measurements of the shininess of supercalendered book, waxed, glassine, or other shiny papers measuring more than 75% gloss by this method; nor is it recommended for colored papers. For shiny and/or colored papers use TAPPI Standard T 480 m, Specular Gloss of Paper at 75°.

APPARATUS

The testing instrument shall consist of:

(1) A photometer so fixed in relation to the test specimen at an angle of 57.5° that the amount of polarized light reflected from the test specimen relative to the total amount reflected from the test specimen at an angle of 57.5° may be measured.

(2) A source of diffused illumination consisting of a frosted B lamp, G 18½ bulb, 110 volts, 25 watts, so fixed in relation to the test specimen that the light subtends an angle of 11.25° and falls on the test specimen at an angle of 57.5°.

(3) An opal glass diffusing screen between the source of light and the specimen aperture.

(4) A metal box having the photometer housed in one end, the source of illumination housed in the other end, and an aperture in one side over which the specimen is clamped in a fixed position. The box shall be so made as to exclude all outside light and shall have all of its interior surfaces blackened.

TEST SPECIMEN

The specimens for test shall consist of not less than five pieces of paper cut from different portions of the test sample, secured by the official sampling method so as to be representative of it. These shall be of sufficient size to extend beyond all sides of the aperture of the metal box. The test specimens shall be clean, free from wrinkles and folds, shall not be exposed for any considerable length of time to extreme atmospheric conditions, and the areas actually tested shall not have been touched with the fingers.

PROCEDURE

The test specimen shall be firmly clamped in position so that its sides extend beyond all sides of the aperture of the metal box. If the paper is translucent, a sufficient number of test specimens shall be stacked one upon another so that no incident light can penetrate entirely through them. Two tests shall be made on each side of not less than five specimens, one test in the machine direction of the paper and one in the cross direction. The test results shall be recorded in degrees to the nearest 1/5°. The average degrees of gloss shall be computed and converted to per cent gloss by the formula:

$$\text{Per cent gloss} = \cos 2\,(60° - \text{degrees gloss}) \times 100$$

Duplicate determinations shall agree within 0.5% gloss.

REPORT

The results shall be reported in percentage gloss to the nearest 0.1%. The average, maximum, and minimum gloss shall be reported. Where required, in order to show the difference in finish between the two sides of the paper, the gloss of each side shall be reported separately.

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Specular Gloss of Paper at 75°

TAPPI Tentative Standard T 480 m-51

(THIS STANDARD IS UNDER THE JURISDICTION OF THE OPTICAL PROPERTIES COMMITTEE)

Gloss is the geometrically selective reflectance of a surface responsible for its shiny or lustrous appearance. (Surface reflectance is commonly at a maximum in or near geometric directions in which a mirror would reflect light).

This is a method for measuring the specular (mirror) reflectance of paper at 75° (15° from the plane of the paper). Specular glass at 75° is widely used as a partial measure of the surface quality and shiny appearance of paper. It is not, however, a measure of mirror quality and should not therefore be used for cast-coated and other papers reading 80 or higher. Its chief application is to coated book, waxed, and other shiny papers, white or colored, which measure from about 15 to 70 (*1*).

With white papers reading 15 or lower, TAPPI Standard T 424 m, based on the Ingersoll Glarimeter, is recommended because of the better separation given in this gloss range of ledger papers, writing papers, newsprint, and the like. In this low-gloss range, the contrast-gloss instrument, which employs a 57.5° angle, gives a good measure of the tendency of papers to glare when observed under improperly distributed illumination. Tests (*2*) show the existence of partial, but not perfect, correlation between gloss values from the two methods.

APPARATUS

The apparatus shall consist of a light source and a positive lens giving an incident beam of rays, means for holding the specimen of paper flat in position to receive this beam of rays, and a light receptor located to receive certain of the rays reflected by the specimen. These three parts shall be combined in a rugged instrument having the geometric, spectral, and photometric characteristics specified below. The general arrangement of these parts shall be as indicated in Fig. 1.

Geometric Conditions

The axis of the incident and reflected beams shall each intersect the center of the specimen exposure area at an angle 75° from the perpendicular, and the axis of the reflected beam shall be the direction of mirror reflection of the axis of the incident beam. With a front-face mirror in specimen position, an image of the source shall be formed in the center of the receptor window. This image shall be not more than one fourth the width of this receptor window in the plane of measurement (same as plane of Fig. 1), nor more than one half the width of the window across the plane of measurement. All critical dimensions, expressed in arbitrary units, shall be as given in Fig. 1. The reference distance, given as unity (1.00) in the figure is that distance between the center of the receptor window and the center of the specimen. Actual dimensions may be obtained by multiplying the foregoing actual distance (which may be any desired value) by the distances given in arbitrary units. Dimensions not shown may be varied at will. The receptor window shall be round and its circular edge shall be beveled on the side facing the receptor.

Note: To check this image for centering and size, cut a piece of cross-section paper to just fit in the receptor window and place it there with a piece of Scotch tape. It should be possible to adjust the lens position until the image appears well centered and distinct in the center of the cross-section paper.

The aperture stop (B-B in Fig. 1), shall be adjacent to the lens and shall be centered on the optic axis of the illuminating system. It shall be not more than 0.15

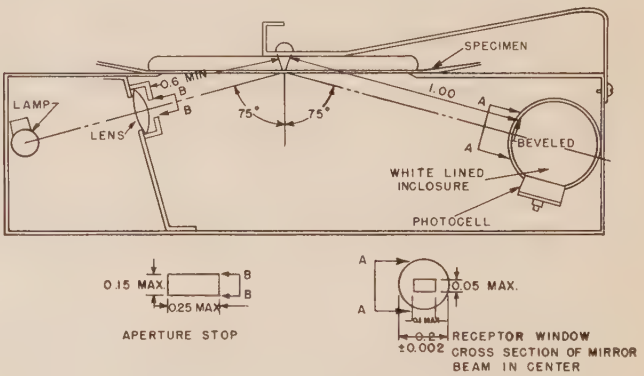

Fig. 1. Diagram of glossmeter showing interior with essential relative dimensions

arbitrary units in the plane of the drawing, nor more than 0.25 across the plane of the drawing, as shown. There shall be no other stops or diaphragms which interfere with incident rays, nor with reflected rays directed toward the receptor stop.

Spectral Conditions

The instrument shall give numerical results not significantly different from those that would be given if its source were I.C.I. Standard Illuminant A and its receptor had the spectral response of the I.C.I. luminosity function.

Photometric Mechanisms

The light-measuring mechanism, such as a photocell and electric meter, shall give a numerical indication which is proportional to the amount of light passing the receptor window to within ±1% of the full-scale amount. Care must be taken to avoid nonuniform weighing of accepted rays due to nonuniform response to the sensitive area of the photocell. A small white-lined sphere to diffuse the accepted light before it reaches the photocell is the means shown in Fig. 1 to

avoid nonuniform weighing, but there are other satisfactory devices meeting this requirement. In general, a two-photocell null method of measurement is preferable to a single photocell connected with an electric meter, but in every case the measuring device must be one that operates accurately (*3*, *4*) with the photocell and light source employed.

Gloss Standards

The fundamental specular-gloss standard is the ideal, completely reflecting, perfect mirror which, for this method, is assigned the value 383. A flat clean, and continuous surface having a refractive index of 1.54 for the sodium D line may be shown by the Fresnel equation (*5*) to measure 100 on this scale. For a working standard, a clean plaque of polished black glass, for which 75° specular reflectance has been computed from refractive index, is recommended.

Except where there are facilities for checking the alignment requirements for the geometric conditions already specified, there shall be an additional standard of intermediate gloss with each instrument. Low-gloss, bathroom wall tiles, each flat to ±0.02 inch in a 4-inch square area, are suitable for this purpose. Each tile must be calibrated on a precise gloss meter which is carefully checked for compliance with the requirements of this specification.

Keep standards clean and away from gritty dirt which might scratch them. Never put them face down on a surface which may be dirty. Both high and intermediate-gloss standards should be periodically cleaned with mild soap, warm water, and a brush. They should be rinsed with water to remove soap and the high-gloss standards should be dried with a clean towel. However, the intermediate standards, which are porous, should be merely blotted and then left to air dry for a day before use.

Note: Both high and intermediate-gloss standards are available from the National Bureau of Standards, Photometry and Colorimetry Section, Washington 25, D. C.; and from the Henry A. Gardner Laboratory, Inc., 4723 Elm Street, Bethesda 14, Md.

TEST SPECIMEN

Specimens for test shall be so selected as to be representative of a test sample secured by the official sampling method. The test specimens shall be conditioned before measurement according to TAPPI Standard T 402 m. Each specimen shall be of sufficient size to completely cover the sample exposure opening of the test apparatus with adequate overlap. Each shall be kept perfectly clean and free from folds and wrinkles; and the area to be tested shall not be touched with the fingers.

Not less than five test specimens shall constitute each test sample.

PROCEDURE

Insert the black-glass standard and adjust the apparatus to give the correct value of gloss for the standard. Insert the intermediate standard and see that the glossmeter reads it correctly. (A correct reading on the black-glass standard alone is no assurance that the glossmeter is in correct adjustment.) Insert each specimen in turn and, unless otherwise instructed, read it for gloss, both in and across the machine direction and on both the felt and wire sides. Make settings on the standards at frequent intervals to insure that the apparatus remains in adjustment throughout the period of operation.

REPORT

The average gloss value for all positions of all specimens and the maximum and minimum values shall be obtained. These shall be reported to the nearest unit if 15 or above, and to the nearest 0.1 unit if below 15.

ADDITIONAL INFORMATION

Diffuse Correction

Traditionally, it is said that the light reflected by a specimen may be divided into one part reflected specularly in the direction of mirror reflection (associated with gloss) and another part reflected diffusely in all directions (associated with lightness on the white-gray-black scale). According to this traditional picture, a gloss reading obtained by exposing a photocell to the light reflected in the direction of mirror reflection needs always to be diminished to compensate for that amount of the measured light attributable to diffuse reflectance. Although it is seldom possible in practice to analyze reflected light according to the traditional picture and say exactly what part is diffuse and what part is specular, it is nevertheless frequent practice where gloss values of light and dark surfaces are being compared to "correct" (diminish) specular gloss settings for diffuse reflectance. Actually the magnitude of this correction for the present test method is so small that it will generally be negligible. It is equal to the diffuse reflectance of the test surface expressed on the scale on which a perfect white is 1.0; hence can never be greater than 1.0. For example, in comparing the gloss of two low-gloss inks, one light yellow and one dark blue, an investigator might want to apply corrections in order to keep the resultant gloss value for the yellow from being spuriously high relative to that for the blue. The diffuse corrections in this case would be about 0.6 for the yellow, 0.1 for the blue.

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SECOND COATING CONFERENCE
TECHNICAL ASSOCIATION
OF THE
PULP AND PAPER INDUSTRY

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Instrumentation Studies LXI. Erasability of Paper.

I. A General Discussion of Erasability

J. A. VAN DEN AKKER

Definitions and statements concerning erasability obtained from recognized technical people in the writing paper industry are presented and discussed. The various factors which are called into play in a judgment of erasing quality are enumerated and discussed, with particular reference to the probable success or failure of control or measurement of the factors. It is concluded that a concise, yet adequately specific, definition of erasability is not to be expected. The phenomena of erasure should be studied with research equipment, and such a study may form the basis for a later, detailed definition of erasability.

Although commonly evaluated by the manufacturers and consumers of writing papers, erasability is not measured in the sense that one can place paper in some sort of instrument and obtain a number that is accurately descriptive of the erasing quality of the paper. In the past, the erasing quality of paper has been judged subjectively through erasure performed by hand, with various kinds of erasers, and a number of factors have been involved in the judgment. These factors include the ease or difficulty of abrading the paper, the appearance of the erased area, and the writing quality of the erased surface after one or more erasures. It is, therefore, not surprising that an instrument for testing erasability which would generally be considered satisfactory has not been created. Nor will it be possible for such an instrument to be developed before a clear, generally acceptable definition of this property has been evolved.

Several erasing machines have been devised which eliminate the human variability in making hand erasures (*1–4*). These machines have provided an important step in the direction of objective evaluation of erasure but, unfortunately, they are only machines—not measuring instruments; such machines subject the paper to a more or less standardized abrading action—they cannot measure the effects on the paper of erasure. The difficult problem of evaluating these effects is recognized by everyone in the writing paper industry. Accordingly, it was suggested several years ago by the Subcommittee on Instrumentation Research (A.P.P.A.) that erasability be made the subject of an instrumentation research. As a beginning, definitions of erasability were obtained from several of the leading technical persons in the writing paper industry. Before the study could be initiated, the war necessitated a change in the research program, and further work on erasability was delayed until early in 1947.

DEFINITIONS OF ERASABILITY

With the exception of the first three definitions given below, all the following comments on erasability have been made by recognized technical individuals of long experience in the writing paper industry.

"That property of a sheet which permits writing on the sheet to be removed by erasure and which resists erosion by erasure. After erasure, the sheet should present a satisfactory appearance on the erased area and of the writing on the erased area." (The Dictionary of Paper. American Paper and Pulp Association.)

"F-3a. Lines of varying widths and of black waterproof drawing ink will be drawn upon different portions of the sample and carefully inspected through a magnifying glass; there shall be no indications of spreading and 'feathering.'

"F-3b. Not sooner than 5 minutes after the lines have been drawn they will be erased with a steel eraser or razor blade and the erased surfaces dressed down with an eraser of the 'Ruby' type. Ink lines (as described in subparagraph F-3a) will be drawn upon the erased surfaces and erased as described for the first lines. Ink lines will again be drawn on the erased surfaces and erased for the third time. Ink lines will again be drawn upon the erased surfaces and examined through a magnifying glass; there shall be no indications of spreading and 'feathering.' (Federal Specifications for Drawing Paper, UU-P-226.)

"The term 'erasing quality' shall be construed to mean the retention of good writing quality and appearance of surface after erasure of ruled lines or written characters. (From the specifications and standards of a large life insurance company.)

"Apparently the term erasability may imply one or more distinct features or properties to different paper users, depending upon the grade of paper involved. For example, on typewriter paper the feature erasability would probably imply the ease with which typewritten matter is removed from the sheet. In some cases, although not always, the appearance of the erased portion is also taken into consideration. The erasability may also imply the erasing of pen and ink lines as in the case of ledger papers and drawing papers. Here the actual speed of erasure is important together with the amount of abrasion on the sheet, as well as the suitability of the erased portion for rewriting. You are probably familiar with the erasing tests on page 3 of the Federal Specifications for Drawing Paper, UU-P-226. Most high-grade ledger paper manufacturers use this same test although it is very difficult to grade papers numerically on this basis.

"Aside from the details of the type and method of applying the impression and the methods of removing the stain, it would seem that the erasability factor could

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at least be subdivided into three distinct subfactors; i.e., the actual ease of removing the impression from the sheet, the cleanliness or the amount of abrading on the erased portion, and third the suitability of the erased portion for rewriting.

"We have tried several methods of evaluating this property which were based on mechanical means of abrading the sheet. However, we were finally obliged to abandon the mechanical method and go to actual manual erasure.

"Our experience is that by 'erasability' most users refer to the condition of the surface of the paper after erasure and the suitability of this surface for rewriting and retyping.

"We have known some users who, by this term, meant the ease with which the characters could be erased. However, we believe that the statement in the first paragraph is in accord with the idea generally accepted.

"We have had two types of complaints on erasure and they are quite different.

"The first pertains to drawing paper. Here we try to produce a sheet that is physically hard all the way through and as well sized as we can make it. The hardness is put there so that the paper will resist abrasion by erasure as much as possible, and the sizing is put there so that when a new ink line is drawn on the erased portion it will not feather. A certain company of New York has told me they have seen a good drawing paper take as many as 15 erasures in the same spot and still not feather when inked in. If this was done, though, I believe that the man who did the erasing did a very nice job. It is the practice in most offices to use a steel eraser and then burnish off with a soft eraser, while others use a rather hard sand eraser and burnish off with a soft eraser. Offhand, it looks as if the ability of the sheet to withstand feathering is going to be tied in with its erasability.

"Now the other type of erasure that I have reference to is entirely different. We have had a few complaints from customers who are using ordinary bond papers and we find they do not like a paper from which it is too hard to remove ordinary typewriter ink. There is, as you know, a paper on the market made according to a patent taken out by J. G. Callan. This paper has a coated casein surface. If the coating is a little bit too thick, it is possible to make the type ink erase so easily that it will smudge. Careful control of the film or the thickness of the film is essential.

"This is a very hard question to answer definitely as erasability will largely depend on the use to which a given paper is intended. Our experience on erasability has been confined chiefly to drawing, tracing, and bond papers. We distinguish between erasability with pencil and with ink. By ink we mean waterproof drawing ink exclusively."

Ink Erasibility

This property has been tested by us for many years by drawing ink lines on the surface of the paper, erasing a portion of the lines with a steel knife, and redrawing new lines over the erased spot at an angle to the original lines. This procedure is then repeated a number of times in accordance with the degree of erasability required in a given paper. We are giving you herewith an extract from the current Government Specifications on Drawing Paper, UU-P-226, which gives a detailed description of how the erasing test on drawing papers is to be made.

Pencil Erasability

For drawing papers the requirement is that it must be possible to erase pencil lines drawn with reasonable firmness without leaving any impressions in the paper and without causing any linting of the surface.

On tracing papers, the same requirement applies, but in addition the erased lines must not leave any traces of "ghosts" which will show on a blueprint paper.

[Federal Specifications for Drawing Paper, UU-P-226, given at this point (see the second quotation given in this section).]

This test [Fed. Spec. UU-P-226] has always been a very questionable one as far as we are concerned and it usually has been up to the judgment of the person testing this property as to its meeting the specification.

"With regard to erasability, I would define it as: 'That quality of a paper which allows the removal by erasing of written, typed, or drawn lines or characters from its surface without seriously impairing the surface for further typing, writing, or drawing.' In the case of pen and ink work the common objection to erasing quality seems to be when feathering occurs in writing over erased areas. In typing and pencil work the objection seems to come when the rewrite is smudged due to loose fibers in the erased area. Apparently erasure is a result of two factors, how much erasing is required to remove the lines or characters and the condition of the sheet after erasing. In pen and ink work it is largely a matter of satisfactory sizing throughout the sheet structure.

"The term 'erasability' as applied to bond, index, and ledger papers designates the property of a sheet of paper to withstand erasure and the use of writing ink satisfactorily after erasure without spreading or penetration of the ink. In some specifications and for certain uses it is required that a sheet of paper be so sized that it will withstand as many as two or three erasures in the same spot and yet possess good writing quality after each erasure. Specifications sometimes require that tests be made with a steel eraser. We seldom use the steel eraser; however, we usually make our erasure tests with a rubber eraser containing a very fine emery."

DISCUSSION

The foregoing definitions are in agreement on several aspects of erasability. Although there is room for further remarks on the subject, it would seem that "saturation" of the chief ideas concerning the property has been attained and that little would be gained through a more extensive quest for definitive statements. The subjective nature of the currently employed erasability tests and the absence of objective means for evaluating the erasing quality of paper are implicit in all the definitions.

Certain conclusions may be based on the material presented in the foregoing section:

1. The procedure followed and the materials employed in judging the erasability of paper depend on the function of the paper. Thus, if a drawing paper is to be tested, lines are drawn upon the paper with waterproof black ink; erasure is performed with a steel eraser,

followed by burnishing with a suitable rubber eraser, and ink lines are redrawn over the erased portion; this process is repeated a number of times, and the appearance of the paper and of the final ink line is carefully observed. By implication, on the other hand, papers designed to carry characters written with ordinary writing ink, pencil, or typewriter are erased a single time with a rubber eraser and the final impression of ink, pencil, or typewriter and the paper are inspected for appearance.

2. A most important factor in the judgment of erasing quality is the appearance of both the paper and the rewritten or redrawn characters in the erased area. The paper is inspected for change in appearance, the characters are examined for spreading, feathering, and smudging.

3. Ease of erasure is an important factor.

In view of the first conclusion given above, it would appear to be impossible to develop a single, concise definition of erasability that would possess sufficient specificity to be really useful. As a general rule, definitions of properties or of quantities can be both concise and specific when all factors concerned are individually amenable to scientific definition or measurement. We are here concerned with a set of factors which are individually either difficult to control or not completely amenable to measurement. Some of these are the following.

Appearance of the Paper

It is not yet possible to measure the appearance of paper. A moment's consideration will indicate that change in appearance is of importance in the problem of evaluating erasability, because the observer, consciously or unconsciously, compares the appearance of the paper in the erased area with that of the surrounding unerased paper. Certain, but not all, aspects of the change in appearance can be measured; the change in color may be determined with spectrophotometric equipment (this determination will include any darkening resulting from decreased opacity in the erased area); the change in gloss for one or more angles of illumination may be measured with suitable photoelectric equipment. The degree of development of fuzz has been determined with a photoelectric device designed for the purpose (Report No. 15) (*5*), but the measurement is one calling for unusual skill and is difficult to standardize; it is possible, although not yet proved, that a suitable measurement of gloss would correlate with fuzz development. The appearance of fine grooves in the erased area (produced by the eraser) cannot be measured. An early observation made in the current study is the reduction of cockle by the action of the eraser. Thus far, no objective means for measuring cockle has been developed, and it is clear that a change in the degree of cockle will modify the appearance of the paper.

The Eraser

A standardized abrading material has not yet been developed. Characteristics of importance in standardization are abrasiveness against paper, point-to-point uniformity of abrasiveness of the eraser, fouling or self-cleaning tendency of the abrading material, and tendency of the abrading material to impart color to the paper. The use of the steel eraser (razor blade or razor-sharp knife) is clearly an art and no means are at hand for the mechanical simulation of that art.

The Ink, Pencil, and Typewriter

Aqueous drawing and writing inks cover a wide range in penetrability. Certain waterproof drawing inks do not penetrate well-sized paper deeply and leave a relatively heavy deposit of pigment on the surface of the paper, whereas there is at least one writing ink in fairly wide use which has apparently been designed to defeat the sizing in paper, because it penetrates into paper rapidly and relatively deeply. Through careful control of chemical composition, it is possible to make a "standard ink," and such standard inks have been employed in studies of writing quality and erasability. It should not be assumed, however, that the relative order of receptivity of a series of papers is the same for all aqueous inks. It is known, for example, that the ranking of papers as to their receptivity to distilled water is quite different from that established with writing ink. In view of the differences between writing inks with regard to colloidal nature and acidity, we should not assume that the ordering of papers according to receptivity to a "standard ink" will be the same as that for some other standard ink. We know, of course, that the amount of fiber which must be removed by erasure to expunge a character depends upon the depth of penetration of the ink. Thus, the ranking of papers based on an evaluation of erasability might be expected to depend upon the choice of a "standard ink." In the interest of definiteness, a certain ink should be adopted, but adoption should be based on a study of the relative receptivities of the various kinds of writing paper to the several kinds of writing ink in common use; ideally, that ink should be chosen which gives rise to a ranking of papers with regard to receptivity which is most representative of the rankings observed with the several inks.

The manner of impression of a drawing or writing ink on paper is an important factor in erasability. The draftsman's ruling pen has been most commonly employed in improved methods for judging erasing quality. Ruling pens vary a great deal in sharpness, and it seems certain that the force of application of the pen to the paper has varied widely in the hands of different operators. In certain laboratories devices have been constructed for the mechanical operation of ruling pens, and it might be possible to standardize the various factors involved. Schur and Levy (*6*) have described a mechanical pen which appears to be both satisfactory in operation and amenable to standardization.

The standardization of a graphite pencil with regard to composition and physical properties of the "lead" would not appear to be the only, or most formidable, factor in the evaluation of erasing quality with respect to penciled lines or characters. The amount of graphite transferred to the sheet would be expected to be a function of the abrasiveness of the paper (e.g., "tooth" of the surface) to graphite and the local pressure under the point of the pencil, the latter being a function of the size and shape of the point, the force of the pencil against the paper, the compressibility of the paper, the nature of the backing beneath the paper, and so on. A complicating feature of the problem of evaluating the erasability with regard to penciled lines or characters is the mechanical grooving of the paper by the pencil.

The evaluation of erasability of paper with regard to typewritten characters involves an array of factors different from those discussed above for inks and pencil. Insofar as receptivity of the paper is concerned, we are here dealing with an oilbase ink. It appears to be well established that the commonly employed methods of sizing to impart water resistance to paper do not have appreciable influence on the receptivity of paper to oils, except where the sizing may reduce the effective pore diameter and, thereby, influence the permeability of the paper. It is clear that the typewriter ribbon must be standardized with respect to such factors as the ink content, the nature and composition of the ink, and the physical properties of the textile ribbon. The design of the type face and the intensity of impact must be controlled (a number of persons have employed electric typewriters to obtain some degree of control of the latter factor), and the hardness of the platen must be held constant. As in the evaluation of erasability with regard to penciled matter, the mechanical impression in the paper produced by the type face is a complicating aspect.

The appearance of lines and characters of ink, pencil, or type may not, as yet, be evaluated in any way except through visual inspection and personal judgment. When an experienced observer performs an inspection, a complex of mental and psychological responses is invoked. The problem of evaluation is not essentially unlike that of judging a work of art, in spite of the large gap in esthetic appeal between written characters and, for example, a painting. It may never be possible with simple instruments to measure such detractions from perfection as feathering of an ink line, small but noticeable uninked spots in a line, and variability of color along the line. Spreading of ink can be measured, as suggested by Schur and Levy (*6*). It is possible that the judgment of feathering can be rendered more objective through the development of procedures for counting the number of flaws per unit length of line, and through the use of pictorial standards, which may be of aid in classifying the flaws with respect to size.

Ease of Erasure

There is some expectation that ease of erasure can be more readily and accurately measured than any other factor involved in erasability. Indeed, the straightforward nature of this factor has apparently led a number of persons to attempt to evaluate erasability through measurement of the loss in weight or thickness of paper per abrading stroke or per unit time of abrading action. There is no question that, all other factors being the same, the ease of erasure is an important attribute of erasability. It is very important, however, that one avoid the temptation to place undue emphasis on ease of erasure. It is clear that the amount of fiber which must be removed in erasure is that which is necessary to expunge the written or typed character. Accordingly, the ease of erasure should be evaluated in terms of the time (for a given mechanism), or the number of strokes, or the amount of energy required *to remove the character*.

THE NEED FOR STUDY OF ERASABILITY

The discussion presented in the foregoing section indicates that erasability is not the kind of property which may be defined in a manner that is both concise and adequately specific to be useful. Prior to the development of suitable, detailed definitions, the various phenomena of erasure should be carefully studied. Such a study might well employ research equipment that is quite different in nature from the apparatus which will eventually be developed on the basis of the study for the laboratory evaluation of erasability. The research equipment and the observations presented in Parts II and III of this report represent the exploratory phase of our study of the phenomena of erasure.

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Grateful acknowledgment is made of the kindness and effort of several technical people in the paper industry in discussing and defining "erasability."

Paper and Packaging Specifications

U. S. GOVERNMENT SPECIFICATIONS

Following is information from B. W. Scribner, National Bureau of Standards, Chairman, Technical Committee on Paper and Paper Products, Federal Specifications Board, on revised Federal specifications of recent issue:

UU-P-158a, Paper, Carbon, Typewriter, Black, Price 5 cents.

Three weights and two grades of paper are now specified. The stock of the higher grade is cotton or linen fibers. The stock of the lower grade is free from wood fibers for lightweight paper, and free from groundwood pulp for heavyweight paper. Changes have also been made in the number of impressions and the number of manifold copies required.

UU-N-106b, Napkins, Table, Paper, Price 5 cents.

The important changes are the addition of another grade containing a minimum of 50% chemical wood fibers, and requirements for absorptiveness and brightness that are intended to ensure satisfactory softness and color, respectively. The revision was developed with the assistance of the Technical Committee of the Paper Napkin Association.

UU-C-201b, Cardboard (Bristol, Manila, and Railroad), Price 5 cents.

The revision was made primarily to bring the specification into accord with the new form for Federal specifications. Only two important changes were made in the technical requirements. These are that the stock on manila board be free from groundwood pulp, and that the liners of railroad board may contain 25% of groundwood pulp.

UU-P-264a, Paper, Concrete-Curing, Waterproofed (Kraft), Price 5 cents.

The important changes in the technical requirements are a requirement that both plies shall be treated for scuff resistance, and that the reinforcing cords or strands of fibers average not less than 20 per foot in each direction. The Technical Committee of the Waterproof Paper Manufacturers Association assisted in the preparation of the revision.

(Copies of Federal specifications and the Federal Specifications Index may be obtained on application accompanied by postal note, money order, coupon or cash to the Superintendent of Documents, Government Printing Office, Washington 25, D. C. The price of the Index is 35 cents.)

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Second Coating Conference, Statler Hotel, Boston, Mass., April 18–20, 1951
- General Chairman—J. H. Heuer, St. Regis Paper Co., Deferiet, N. Y.
- Program Chairman—Alfred D. Bonanno, Champion-International Co., Lawrence, Mass.

Sixth Engineering Conference of the Pulp and Paper Industry, General Oglethorpe Hotel, Wilmington Island, Savannah, Ga., October 15–18, 1951
- General Chairman—George H. Pringle, Mead Corp., Chillicothe, Ohio
- Local Arrangements Chairman—James R. Lientz, Union Bag & Paper Co., Savannah, Ga.

LOCAL SECTIONS

Ohio Section:
- March 8, 1951, Manchester Hotel, Middletown, Ohio.
- April 10, 1951, Manchester Hotel, Middletown, Ohio.
- May 10, 1951, Manchester Hotel, Middletown, Ohio.

Pacific Section:
- April 13, 1951, Longview, Wash.

New England Section:
- April 18–19, 1951, Statler Hotel, Boston, Mass.

Lake Erie Papermakers and Converters:
- February 16, 1951, Hickory Grill, Cleveland, Ohio.
- March 16, 1951, Hickory Grill, Cleveland, Ohio.
- April 20, 1951, Hickory Grill, Cleveland, Ohio.
- May 18, 1951, Hickory Grill, Cleveland, Ohio.

Delaware Valley Section:
- March 22, 1951, Engineers Club, Philadelphia, Pa.
- April 26, 1951, Engineers Club, Philadelphia, Pa.
- May 24, 1951, Engineers Club, Philadelphia, Pa.

Chicago Section:
- March 19, 1951, Chicago Bar Assoc., Chicago, Ill.

Metropolitan Group, Empire State Section:
- March 6, 1951, Fraunces Tavern, New York, N. Y.
- April 3, 1951, Fraunces Tavern, New York, N. Y.

Northern Group, Empire State Section:
- March 8, 1951, Woodruff Hotel, Watertown, N. Y.
- April 12, 1951, Woodruff Hotel, Watertown, N. Y.
- May 10, 1951, Woodruff Hotel, Watertown, N. Y.

Eastern Group, Empire State Section:
- February 16, 1951, Milfranks Restaurant, South Glens Falls, N. Y.

Lake States Section:
- March 13, 1951, Stevens Point, Wis.

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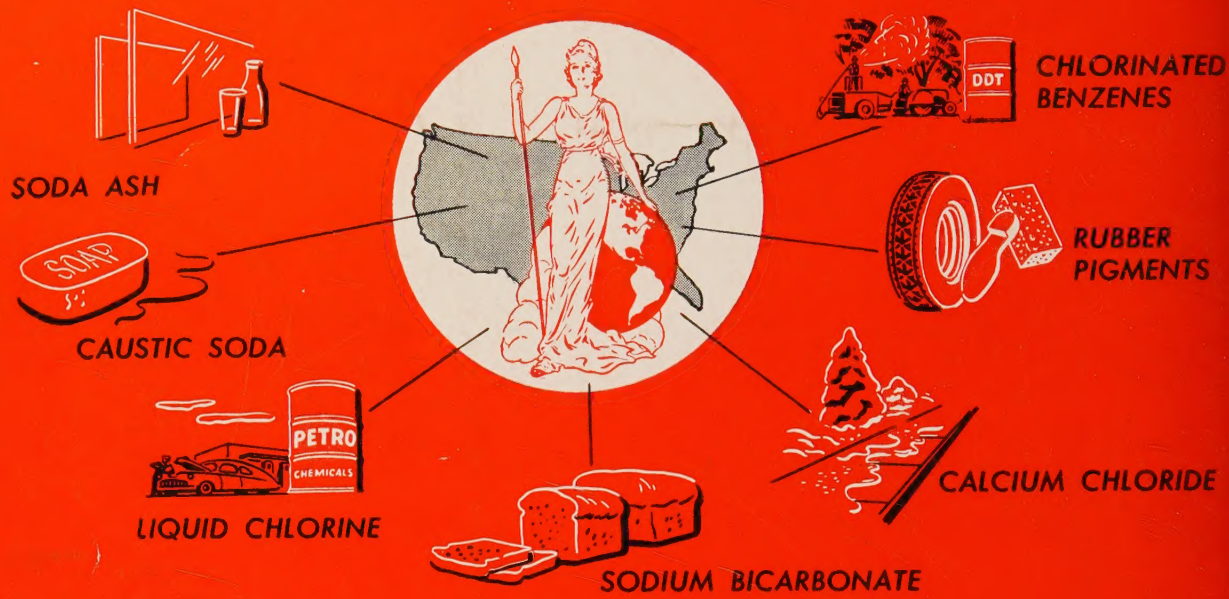

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